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QUARTZ LAMP RADIANT HEAT BRAZING
OF
LARGE REFRACTORY METAL HONEYCOMB SANDWICH PANELS

INTERIM ENGINEERING PROGRESS REPORT NR-IR-7-937a(||)
10 October 1962 - 10 January 1963

Fabrication Branch
Manufacturing Technology Laboratory
Aeronautical Systems Division
Air Force Systems Command
United States Air Force
Wright-Patterson Air Force Base, Ohio

ASD Project Nr. 7-937a

295 703

(Prepared under Contract AF33(657)-8910 by the Northrop Corporation, Norair Division, Hawthorne, California, D. B. Hugill, B. Gaiennie')

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FOREWORD

THIS INTERIM ENGINEERING PROGRESS REPORT COVERS THE WORK PERFORMED UNDER CONTRACT AF 33(657)-8910 FROM 10 OCTOBER 1962 TO 10 JANUARY 1963. IT IS PUBLISHED FOR TECHNICAL INFORMATION ONLY AND DOES NOT NECESSARILY REPRESENT THE RECOMMENDATION, CONCLUSIONS OR APPROVAL OF THE AIR FORCE.

THIS CONTRACT WITH THE NORTHROP CORPORATION, NORAIR DIVISION, HAWTHORNE, CALIFORNIA, WAS INITIATED UNDER MANUFACTURING METHODS PROJECT 7-937A. IT IS BEING ACCOMPLISHED UNDER THE TECHNICAL DIRECTION OF MR. B. E. PRICE OF THE FABRICATION BRANCH (ASRCTF) MANUFACTURING TECHNOLOGY LABORATORY AND MR. P. P. PLANK, STRUCTURAL RESEARCH BRANCH (ASRMDS-21) FLIGHT DYNAMICS LABORATORY, AERONAUTICAL SYSTEMS DIVISIONS, WRIGHT-PATTERSON AIR FORCE BASE, OHIO.

CLOSELY RELATED EFFORTS ARE COVERED UNDER ASD PROJECT 7-937 "Manufacturing Methods and Design Procedures of Brazed Refractory Metal Honeycomb Sandwich Panels".

MR. D. B. Hugill of Norair's Manufacturing Research and Development Group was the engineer in charge. Major contributions to the effort were made by Messrs. P. W. Warren and R. A. Bridwell of the Manufacturing Research and Development Group; Messrs. J. D. Revell, D. R. Apodaca and J. L. Malakoff, Structures Analysis; and Mr. A. H. Freedman, Material Sciences Laboratory. Contractor has assigned Report NOR 63-13 for internal identification.

This Project has been carried out as part of the Air Force Manufacturing Methods Program. The primary objective is to develop, on a timely basis, manufacturing processes, techniques and equipment for use in economical production of USAF materials and components. This program encompasses the following technical areas:

REFRACTORY METALS, ROLLED SHEET AND FOIL, COMPONENT FABRICATION, FORMING, JOINING (BRAZING, WELDING, DIFFUSION BONDING), METALS REMOVAL, PROTECTIVE COATINGS, CLEANING.

Your comments are solicited on the potential utilization of the information contained herein as applied to your present or future production programs. Suggestions concerning additional Manufacturing Methods Development required on this or other subjects will be appreciated.

ABSTRACT

QUARTZ LAMP RADIANT HEAT BRAZING OF LARGE REFRACTORY METAL HONEYCOMB SANDWICH PANELS

D. B. HUGILL

ET AL.

NORTHROP CORP., NORAIR DIVISION

THIS PROJECT IS PLANNED TO DEVELOP MANUFACTURING METHODS AND DESIGN CRITERIA FOR REFRACTORY METAL BRAZED SANDWICH STRUCTURES OF SUFFICIENT SIZE TO BE PRACTICAL FOR AEROSPACE VEHICLES. THE QUARTZ LAMP RADIANT HEATING SYSTEM WILL BE USED. INVESTIGATION HAS DEMONSTRATED THAT DIFFUSION BONDED HONEYCOMB CORE CAN BE PRODUCED AND THAT SUCCESSFUL BRAZING CAN BE ACCOMPLISHED UNDER TIME, TEMPERATURE, AND INERT GAS ATMOSPHERE CONDITIONS REPRESENTATIVE OF THE QUARTZ LAMP RADIANT BRAZING SYSTEM.

Thermal flux data are presented for a hypothetical re-entry pattern at 500,000 foot altitude, -7° flight angle at escape velocity. It is shown that maximum surface temperatures of 3000°F are reached under such a re-entry pattern.

Sound diffusion bonds for honeycomb core manufacture can be made from 2 mil D-36 and TZM foil using a 30 micro-inch or greater deposit of titanium as an interface agent. No recrystallization of the TZM alloy occurs from the bonding cycle.

RECRYSTALLIZATION STUDIES ON TZM SHOW THAT A NORTOBRAZE CYCLE OF 2400°F WILL LIMIT RECRYSTALLIZATION EFFECTS TO THE EXTENT THAT SATISFACTORILY DUCTILE STRUCTURAL PANELS WILL RESULT.

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1. INTRODUCTION

The purpose of this program is to utilize data which has resulted from closely related ASD projects to establish and develop the design, manufacturing methods and processes and the fabrication techniques for quartz lamp brazing of large refractory metal honeycomb sandwich panels. The panels are to be suitable for application on aerospace vehicles as either hot structure or heat shields. Insofar as possible the materials chosen, the panel design, and brazing and processing criteria will be predicated on the related project results.

The program is now in Phase I of a three-phase program: Phase I, preliminary Studies; Phase II, Panel Manufacturing and Testing; and Phase III, Test Analysis.

Phase I consists of preliminary analysis and studies necessary for the design, manufacture and testing of large honeycomb panels and includes: (1) materials evaluation, selection and procurement; (2) preliminary structural design and analysis studies to establish test panel configuration and to predict thermal response; (3) determination of braze alloys to be used on this program; (4) manufacturing process studies to develop procedures for fabrication, cleaning and assembly of panel details and for brazing the test panels; (5) manufacture of diffusion bonded molybdenum and columbium honeycomb core; and (6) evaluation of envelope materials and protective coatings to function for the duration of the brazing cycle.

PHASE II OF THE PROGRAM CONSISTS OF THE FABRICATION AND TESTING OF THE HONEYCOMB SANDWICH PANELS. DURING THIS PHASE ALL FABRICATION, ASSEMBLY AND TEST TOOLING WILL BE COMPLETED, AND ALL TEST PANEL COMPONENTS WILL BE FABRICATED, ASSEMBLED AND BRAZED. THE COMPLETED PANELS SELECTED FOR ELEVATED TEMPERATURE TESTING WILL BE COATED AND TESTED IN AIR AT TEMPERATURES REPRESENTATIVE OF RE-ENTRY CONDITIONS TO ESTABLISH STRUCTURAL STRENGTH AND THERMAL RESISTANCE. OTHER UNCOATED PANELS WILL BE DESTRUCTIVELY TESTED AT ROOM TEMPERATURE. USABLE PORTIONS OF THESE PANELS WILL BE CUT INTO SPECIMENS AND TESTED AT ELEVATED TEMPERATURES IN A PROTECTIVE ATMOSPHERE TO DETERMINE MATERIAL AND HONEYCOMB PROPERTIES INCLUDING SONIC FATIQUE.

PHASE III CONSISTS OF ANALYSIS AND EVALUATION OF TEST DATA TO DETERMINE PANEL STRENGTH, THERMAL CHARACTERISTICS, DIMENSIONAL STABILITY AND MANU-FACTURING RELIABILITY EMPLOYING THE SELECTED MATERIALS AND DESIGN CONCEPTS. PROCEDURES WILL BE DEVELOPED FOR THE INITIAL DESIGN OF SANDWICH COMPONENTS UTILIZING FORMULAS AND CHARTS PRESENTED IN TERMS OF GENERAL PARAMETERS BASED ON PANEL DIMENSIONS AND MATERIAL PROPERTIES.

2. SUMMARY

This program is now in Phase I with the major portions of panel analysis, re-entry corridor heat flux studies and braze alloy investigation complete. Panel design for both the heat shield and the structural applications is complete and detail drawings have been submitted to ASD for approval.

During this period it has been demonstrated that the use of foil gage titanium interface material for diffusion bonded D-36 and TZM honeycomb core nodes can be successfully replaced with micro-inch deposits of the same material. The resultant bond strength is high and in subsequent brazing operations the micro-film bond should prove superior to the foil gage bond.

Installation of the additional power transformers required for the brazing of panels in Phase II of the program has been delayed pending receipt of Air Force approval for the necessary transformer modifications and installation. Schedules have been revised to initiate the Phase I brazing program without further waiting for a final decision on this matter.

3. PROGRAM REVIEW AND DISCUSSION

A. MATERIALS PROCUREMENT AND EVALUATION

1. PROCUREMENT

The materials to be used in this program are those selected under Contract AF 33(657)-7276 (Reference 1). This selection was established as Columbium alloy D-36 (Cb-10Ti-5Zr) and Molybdenum alloy TZM (Mo-0.5Ti-0.08Zr). Facing sheet gages of .008 and .012 are to be used with honeycomb core foil gage of .002.

The material gages and facing sheet sizes involved precluded obtaining delivery from the prime supply sources of the two alloys and required placement of orders through rerolling mills. Rodney Metals and Fansteel Metallurgical Corporation were selected to supply the D-36 and TZM alloys respectively.

DELIVERY OF .002 FOIL WAS GIVEN HIGHEST PRIORITY (FOR USE IN FABRICATION OF THE HONEYCOMB CORE) FOLLOWED BY SMALL SIZE FACING SHEET GAGES REQUIRED IN TEST BRAZING OF SANDWICHES IN THE NORTOBRAZE FACILITY. BY 1 DECEMBER 1962 NORAIR HAD RECEIVED AND DELIVERED TO HEXCEL PRODUCTS, INC. (FABRICATOR OF DIFFUSION BONDED HONEYCOMB CORE) APPROXIMATELY 8 POUNDS OF D-36 MIL FOIL AND 3.6 POUNDS OF 2 MIL TZM FOIL. DURING DECEMBER DELIVERY WAS MADE ON THE REMAINDER OF THE INITIAL FOIL ORDER FOR D-36, AND APPROXIMATELY 70% OF THE TZM ORDER WAS RECEIVED. THE INITIAL FOIL ORDER FOR EACH ALLOY WAS BASED ON THE NET WEIGHT OF THE FINISHED HONEYCOMB CORE REQUIRED PLUS A NOMINAL SCRAP ALLOWANCE.

PRELIMINARY DESIGN ANALYSIS DEVELOPED DURING THE FIRST QUARTER'S EFFORT REVEALED FACING SHEET GAGES AND CORE DEPTHS SPECIFIED IN THE CONTRACT WOULD RESULT IN LESS THAN OPTIMUM PERFORMANCE FOR THE PANEL SIZES INVOLVED. A STOP ORDER WAS PLACED ON ALL FACING SHEET MATERIALS 14 NOVEMBER WHICH WAS LIFTED 30 NOVEMBER WHEN ASD AUTHORITY WAS GIVEN TO PROCEED WITH THE ORIGINAL SKIN GAGE - CORE DEPTH CONCEPT. THE EFFECT OF THIS ACTION WAS A TOTAL SET BACK OF FIVE-SIX WEEKS FOR READJUSTMENT OF ROLLING SCHEDULES. THESE REVISED SCHEDULES CALL FOR DELIVERY BEGINNING IN JANUARY 1963.

ENVELOPE MATERIALS HAVE BEEN SELECTED AND ORDERED FOR THE TEST BRAZING PORTION OF PHASE I. TITANIUM A-55 WILL BE USED FOR BRAZING TEMPERATURES UP TO 2700-2750°F, CB F82 WILL BE USED FOR ENVELOPE MATERIAL FOR BRAZING IN THE 2800-3200°F RANGE. THESE MATERIALS WILL BE PROTECTED WITH OXIDATION PROTECTIVE COATINGS, DEVELOPMENT OF WHICH IS COVERED IN SECTION C-4 OF THIS REPORT.

From the braze alloy studies conducted in Norair's Material Sciences Laboratory it was determined that the Nortobraze test brazing program should center on three alloys obtainable in foil gage. These are Ti-55A,

TI-13V-11CR-3AL AND L-605 COBALT BASE ALLOY. MINIMUM QUANTITY ORDERS WERE PLACED FOR EACH ALLOY AND DELIVERY IS SCHEDULED TO COINCIDE WITH RECEIPT OF TEST PIECE SIZES OF FACING SHEET MATERIAL. FOIL GAGE OF 1.5 MIL IS DESIRABLE BUT COULD NOT BE OBTAINED IN THE TITANIUM ALLOY OR L-605 WITHOUT A SPECIAL MILL RUN AND ITS ATTENDANT DELAY. Two (2) MIL STOCK WAS ACCEPTED AND WILL BE CHEMICALLY REDUCED IN GAGE IF NECESSARY.

THERMOCOUPLE WIRING FOR PERMANENT INSTALLATION ON THE FACILITY TO BE USED FOR THIS PROGRAM HAS BEEN PROCURED WITH IN-HOUSE FUNDING. THIS INSTALLATION WILL PERMIT SHORT THERMOCOUPLE LEADS FROM THE BRAZING ENVELOPE TO BE PLUGGED INTO TERMINALS ATTACHED TO THE PART HOLDING FRAME.

2. PHYSICAL AND MECHANICAL PROPERTIES

MATERIAL BEHAVIOR

IT IS RECOGNIZED THAT THE UTILIZATION OF MOLYBDENUM AND COLUMBIUM ABOVE 2000°F INTRODUCES MANY PROBLEM AREAS FOR SERVICE LIFE COMPATIBLE WITH THE CHOSEN TEMPERATURE PROFILE. WHENEVER THESE MATERIALS ARE USED BEYOND THIS THRESHOLD LIMIT, THEY BECOME INCREASINGLY SENSITIVE TO CERTAIN FACTORS WHICH ARE NOT YET WELL UNDERSTOOD OR DEFINED, EITHER IN THEORY OR BY MATERIAL PROPERTY DATA. SOME OF THE PHENOMENOLOGICAL EFFECTS DESCRIBING THE BEHAVIOR OF THE PURE METALS ARE SPECULATIVE. THIS FURTHER COMPLICATES BEHAVIOR PREDICTION OF THEIR ALLOYS. SOME OF THE VARIABLES WHICH HAVE A SIGNIFICANT EFFECT ON THE BEHAVIOR OF THESE TWO MATERIALS ARE DISCUSSED IN THE FOLLOWING SECTIONS.

EFFECTS OF THERMAL ACTIVATION

The effects of thermal exposure on the tensile properties of the TZM molybdenum during single or multiple heating cycles can be determined from the curves shown in Figure 1. This information will be used during the experimental portion of the program to help ascertain the residual strength in the material. The thermal exposure data was obtained from Reference 2, and was subsequently reduced for development of the curves shown.

Unfortunately, similar data for the D-36 columbium was not available; nor was it available for both materials at elevated temperature. Unit tests on coupons to produce data in these deficient areas are currently scheduled at Norair. This information should be available prior to the initiation of the experimental portion on panels of this program. A limited amount of work in this area has already been completed for effects of various thermal exposure histories on loss of hardness (DPH) and percent recrystallization of TZM molybdenum and D-36 columbium (Reference 3).

EFFECT OF STRAIN RATE

RATE OF LOADING, OR STRAIN RATE, HAS GREAT INFLUENCE ON THE MECHANICAL PROPERTIES WHICH GOVERN THE BEHAVIOR OF THESE MATERIALS AT TEMPERATURES ABOVE THRESHOLD. AT ROOM TEMPERATURE, AN INCREASE IN THE STRAIN RATE GENERALLY CAUSES ONLY A MODERATE INCREASE IN THE YIELD AND ULTIMATE

STRENGTH. AT ELEVATED TEMPERATURES, HOWEVER, THE EFFECT OF STRAIN RATE BECOMES INCREASINGLY GREATER ESPECIALLY WHEN CREEP DEFORMATION PROCESSES (PLASTIC FLOW) BECOME PREVALENT. THIS CONDITION CAN MARKEDLY AFFECT THE STRUCTURAL RESPONSE OF THE MATERIAL TO RATE OF LOAD APPLICATION DURING A GIVEN RE-ENTRY MISSION. CREEP-TYPE PROCESSES HAVE BEEN DEMONSTRATED TO BE NONLINEAR FUNCTIONS OF THE APPLIED STRESS, TEMPERATURE AND ELAPSED TIME. THIS CONDITION IS FURTHER COMPLICATED BY TEMPERATURE DIFFERENTIALS AND THE PRESENCE OF STRESS CONCENTRATIONS.

THE EFFECT OF CREEP ON THE STRAIN RATE FOR PURE MOLYBDENUM AT 3000°F IS SHOWN IN FIGURE 2. NOTE THAT A TRUE MODULUS OF ELASTICITY AT LOW STRAIN RATES IS NOT EVIDENT DURING THE INITIAL PORTION OF THE CURVE, AS IT IS AT THE HIGHER STRAIN RATES. CLIMAX MOLYBDENUM REPORTED THAT THE SHORT-TIME TENSILE AND LONG-TIME RUPTURE STRENGTHS AT 3000°F ARE ONLY ABOUT 1000 PSI HIGHER FOR TZM THAN FOR THE UNALLOYED FORM. THESE CURVES CAN THEREFORE BE RELATED TO TZM WITHOUT SIGNIFICANT ERROR.

DURING AN ELASTIC DEFORMATION PROCESS, THE STORED ENERGY IS EQUIVALENT TO THE INPUT ENERGY; IF THE ENERGY IS NOT DISSIPATED, THE PROCESS IS RE-VERSIBLE. HOWEVER, IF THE STORED ENERGY IS DISSIPATED BY A RELAXATION PROCESS, THE PROCESS IS IRREVERSIBLE AND PLASTIC FLOW RESULTS. IRRE-VERSIBLE REACTIONS CAN, HOWEVER, BE MINIMIZED BY AVOIDING LOW, STEADY STRAIN RATES AT TEMPERATURES ABOVE THE THRESHOLD TEMPERATURE. THUS, IT IS EVIDENT THAT A LOW STRAIN RATE DURING A RE-ENTRY MISSION WOULD CAUSE A PROFOUND EFFECT ON THE STRUCTURAL RESPONSE. IT IS EVIDENT FROM FIGURE 3 THAT PREDICTING THE QUANTITATIVE EFFECTS OF STRAIN RATE THROUGH A TRANSIENT HEATING SPECTRUM WOULD REQUIRE A RIGOROUS ANALYSIS. (AT THIS WRITING, NO STRAIN RATE DATA WAS LOCATED FOR D-36 COLUMBIUM).

STATIC STRESS

STRESS-TO-RUPTURE TEST DATA CAN BE USED FOR PREDICTING THE LIFE OF A MATERIAL AT THE EXTREME TEMPERATURES. THIS DATA CAN BE FURTHER REDUCED FOR PLOTTING WITH A TIME-TEMPERATURE PARAMETER BASED ON THE ACTIVATION ENERGY RATE-PROCESS THEORY. THIS TECHNIQUE CAN BE USED FOR PREDICTING THE RUPTURE LIFE OF A STRUCTURE SUBJECTED TO EXTREME THERMAL EXPOSURE CONDITIONS. A NOTABLE DISADVANTAGE TO THIS APPROACH, HOWEVER, IS THAT THE TIME-DEFORMATION CHARACTERISTICS, AS NOTED BY CREEP, ARE NOT DEFINED PRIOR TO RUPTURE.

ONLY A LIMITED AMOUNT OF STRESS-TO-RUPTURE DATA WAS LOCATED FOR TZM MOLYBDENUM AND D-36 COLUMBIUM. STRESS-TO-RUPTURE DATA FOR D-36 COLUMBIUM BIUM AT TEMPERATURES ABOVE 2400°F IS NON-EXISTENT. THEREFORE, THE MELTING POINT OF THIS ALLOY WAS USED AS THE REFERENCE TEMPERATURE FOR DEVELOPMENT OF DATA ABOVE 2400°F AND THE CURVE WAS INTERPOLATED BETWEEN THESE TWO TEMPERATURES. CURVES PRODUCED IN THIS MANNER AFFORD A MEANS BY WHICH THE STRUCTURAL RESPONSE OF THE MATERIAL TO RUPTURE CAN BE ESTIMATED AT TEMPERATURES ABOVE 2400°F.

As an approach to predicting the rupture strength in the two alloys (See Figures 4 and 5) the stress-to-rupture data was plotted versus a modified Larson-Miller time-temperature parameter as described by the equation, Θ = T(C + log t), where T is the absolute temperature in degrees Rankine, C is a material constant, and t is the time in hours. These figures were further modified by superimposing the Master Curves for the exposure temperatures (Reference 4). With these curves it is possible to determine directly any time-temperature combination for various thermal exposure conditions.

RECRYSTALLIZATION

WHEN RECRYSTALLIZATION OCCURS AT ELEVATED TEMPERATURE UNDER MODERATE STRESS, THE RESISTANCE TO CREEP DEFORMATION GENERALLY DECREASES QUITZ RAPIDLY, AS ILLUSTRATED IN FIGURE 5. THE INTERCEPT FOR STRESS-RELIEVED MATERIAL TO RECRYSTALLIZED MATERIAL OCCURS AT APPROXIMATELY 2400°F AFTER LIMITED EXPOSURE, FOR TZM MOLYBDENUM. THE RATE AT WHICH RECRYSTALLIZATION OCCURS IS ALSO A FUNCTION OF THE APPLIED LOAD, AS SHOWN IN FIGURE 6. PREDICTING THE INITIATION AND INFLUENCE OF RECRYSTALLIZATION ON THE STRUCTURAL RESPONSE OF COMPOSITE HEAT SHIELDS IS FURTHER COMPLICATED BY OTHER FACTORS AFFECTING THE ALLOY. THESE FACTORS INCLUDE: ALLOY COMPOSITION AND PURITY, TYPE AND DEFORMATION USED IN PROCESSING THE MATERIAL, AND GRAIN SIZES AND ORIENTATION.

EDGEWISE COMPRESSION TESTS

TEST CRITERIA FOR THE EDGEWISE COMPRESSION TESTS OF THE STRUCTURAL PANELS ARE CURRENTLY BEING FORMULATED. DETERMINATION OF THE TEST SCHEDULES FOR THE LABORATORY EXPERIMENTS WILL BE BASED ON THE STRENGTH PROPERTIES OF THE MATERIALS UNDER STUDY.

IN THESE EXPERIMENTS THE LOAD WILL BE APPLIED TO THE PANEL PRIOR TO HEATING, AND WILL BE AUTOMATICALLY ADJUSTED DURING TESTING TO ALLOW FOR THERMAL EXPANSION. THIS WILL RESULT IN UNIFORM PRESSURE LOADING. AUTOMATIC VALVES LOCATED IN THE HYDRAULIC PRESSES WILL BE USED TO PERFORM THIS FUNCTION. END FIXTURES WILL BE USED TO SUPPORT THE PANEL AND TO PREVENT LATERAL WARPAGE OF THE EDGES.

TO PREVENT EXCESSIVE HEAT LOSSES THROUGH TO THE EDGE MEMBERS, A THIN CERAMIC FELT MATERIAL, MADE OF ZIRCONIA OR A SIMILAR MATERIAL, WILL BE PRE-PLACED BETWEEN THE CHANNELS IN THE PANEL AND THE END FIXTURE STEEL BARS. IT IS BELIEVED THAT THIS ARRANGEMENT WILL PROVIDE SUFFICIENT INSULATION TO REDUCE PANEL TO FIXTURE HEAT LOSS TO ACCEPTABLE LEVELS. THE COMPATIBILITY OF EACH PANEL WILL BE DETERMINED IN ACCORDANCE WITH THE RESPONSE OF THE PANEL TO HEATING AND LOADING.

B. DESIGN ANALYSIS

1. THERMAL RESPONSE OF THE STRUCTURE

A. THERMAL RESPONSE CALCULATIONS FOR STRUCTURAL PANEL AND HEAT SHIELD FOR A "TYPICAL" RE-ENTRY TRAJECTORY

THE STATEMENT OF WORK (EXHIBIT "A") OF THE CONTRACT CALLS FOR EDGE LOADING TESTS OF THE STRUCTURAL PANELS FOR TEMPERATURES UP TO 2500°F UNDER THERMAL GRADIENT CONDITIONS WHICH ARE COMPATIBLE. THE HEAT SHIELDS ARE TO BE SUBJECTED TO A 3000°F MAXIMUM TEMPERATURE AND CYCLED THROUGH A TYPICAL RE-ENTRY TEMPERATURE PROFILE, TO BE REPEATED FIVE TIMES. IN ORDER TO MEET THESE REQUIREMENTS THE FOLLOWING PROCEDURE WAS ADOPTED:

- Step (1): Choose a representative trajectory which will yield the desired maximum temperatures.
- Step (2): Compute trajectory parameters for the above trajectory.
- STEP (3): COMPUTE TEMPERATURE-TIME HISTORIES FOR HEAT SHIELD IN-SULATION - STRUCTURAL PANEL ARRANGEMENTS DESIGNED TO ACHIEVE THE DESIRED MAXIMUM TEMPERATURES IN CONJUNCTION WITH THE HEAT FLUX SCHEDULE FROM STEP (2).
- STEP (4): ESTABLISH A HEAT FLUX SCHEDULE FOR PURPOSES OF LABORATORY SIMULATION BASED ON STEP (3), AND ESTIMATE THE STRUCTURAL PANEL TEMPERATURE RESPONSE TO THE PROPOSED HEATING SCHEDULE, CONSIDERING THE LABORATORY ENVIRONMENT.

A DETAILED DISCUSSION OF THESE STEPS FOLLOWS:

STEP (1): Choose a REPRESENTATIVE "TYPICAL" RE-ENTRY TRAJECTORY.

For this purpose a Brief Literature search was made, (Ref. 5...10) AND THE DATA OF REFERENCE (5) (WITH SOME MODIFICATIONS) WAS ADOPTED. IN THIS REFERENCE TRAJECTORIES ARE GIVEN WHICH MINIMIZE THE TOTAL AERODYNAMIC HEAT LEAD, WHILE REMAINING WITHIN THE PILOT ACCELERATION PULSE TOLERANCE. REFERENCE (6) GIVES CONSIDERATION TO BLUNT SWEPT LEADING EDGE LIFTING SURFACE STAGNATION POINT HEAT TRANSFER; HOWEVER, THE MORE CAREFUL ATTENTION TO PILOT ACCELERATION TOLERANCE IN REFERence (5) makes it appear preferable. In Both Reference (5) and (6) THE LIFT VS DRAG RELATIONS ARE BASED ON NEWTONIAN AERODYNAMIC THEORY, WHICH IS DEEMED ADEQUATE FOR THE PRESENT PURPOSE. (SEE REFERENCE (7), Chapter III). Two classes of re-entry trajectories are discussed in Reference (5), 1: A Hypersonic glider for a "once-AROUND-THE-EARTH" FLIGHT STARTING AT ORBITAL SPEED 25,900 FT/SEC AND AN ALTITUDE OF 300,000 FT. 2: RE-ENTRY FROM ESCAPE VELOCITY (35,000 FT/SEC) INITIAL ALTITUDE, 500,000 FT; INITIAL glide path angle, -7° . This trajectory is subjected to a pilot ACCELERATION PULSE TOLERANCE LIMIT. THE ESCAPE VELOCITY RE-ENTRY TRAJECTORY WAS CHOSEN FOR THE PRESENT STUDY BECAUSE IT WAS FOUND THAT THE "SKIP-GLIDE-ONCE-AROUND" TRAJECTORY YIELDS RADI-ATION EQUILIBRIUM SURFACE TEMPERATURES BELOW THE 3000°F LIMIT

WHICH HAS BEEN SET AS THE MAXIMUM FOR THE HEAT SHIELD PANEL DEMONSTRATION. A DISCUSSION OF THE BASIS FOR AERODYNAMIC HEATING CALCULATIONS, AND EQUILIBRIUM TEMPERATURE CONSIDERATIONS FOR THE "SKIP-GLIDE-ONCE-AROUND" TRAJECTORY IS PRESENTED IN APPENDIX A WHICH SHOWS AN ESTIMATED MAXIMUM TEMPERATURE OF 2300°F (WHICH CAN BE REDUCED TO 1900°F WITH A 6-FOOT NOSE RADIUS). THEREFORE, FURTHER TREATMENT OF THE SKIP-GLIDE TRAJECTORY IS NOT PERTINENT AND ATTENTION IS DIRECTED TO THE ESCAPE VELOCITY RE-ENTRY TRAJECTORY AS ONE LIKELY TO PRODUCE THE TEMPERATURE LEVELS OF THE PRESENT RESEARCH PROGRAM AND PROVIDE A RATIONAL BASIS FOR ESTABLISHING TRAJECTORY PARAMETERS VS TIME AND, SUBSEQUENTLY, A TEMPERATURE-TIME HISTORY.

Step (2): EVALUATION OF TRAJECTORY PARAMETERS

THE ESCAPE VELOCITY RE-ENTRY TRAJECTORY IS NOW CONSIDERED FOR A HYPOTHETICAL HEAT SHIELD, INSULATION AND STRUCTURAL PANEL ARRANGE-MENT, WHICH IS DESIGNED TO REACH MAXIMUM TEMPERATURES OF 3000°F AND 2500°F FOR THE HEAT SHIELD AND STRUCTURAL PANEL USING THE AERODYNAMIC HEAT FLUX RATES ASSOCIATED WITH THE VELOCITY ALTITUDE schedule of Reference (5). The velocity and altitude vs time is SHOWN IN FIGURE 7. THE ASSOCIATED ACCELERATION "G" LOAD FACTOR DUE TO THE PULL UP AND THE STAGNATION POINT AERODYNAMIC HEAT FLUX (BASED ON CONSIDERATIONS OF APPENDIX A) ARE PLOTTED IN FIGURE 8. THE "G" LOAD IS ALONG THE RADIUS OF CURVATURE OF THE FLIGHT PATH; HENCE, IT IS PERPENDICULAR TO THE FLIGHT PATH. THEREFORE, ITS EFFECT IN PRODUCING PANEL STRESSES DEPENDS ON PANEL LOCATION. ALSO SHOWN IN FIGURE 7 IS THE STAGNATION PRESSURE VARIATION, SOME FRACTION OF WHICH WOULD REPRESENT THE APPLIED PRESSURE LOADING FOR A PANEL AT THE STAGNATION POINT. THIS MAXIMUM PRESSURE DROPS RAPIDLY WITH DISTANCE AFT FROM THE BLUNT NOSE. FOR SUCH A TRA-JECTORY THE PRESENT HEAT SHIELD DESIGN LOAD OF 1.2 PSI (173 PSF) WOULD BE EXCEEDED WITHOUT SOME VENTING SYSTEM. THE TRAJECTORY OF FIGURE 7 REPRESENTS A MORE GRADUAL LET DOWN THAN THE OPTIMUM TRA-JECTORY OF REFERENCE (5), AND WITH A SUBSEQUENT DECELERATION TO HOLD THE AERODYNAMIC HEAT FLUX SCHEDULE OF FIGURE 8. THIS WOULD REQUIRE A MODIFICATION OF THE ANGLE OF ATTACK SCHEDULE OF TRA-JECTORY OF REFERENCE 7 FOR THE "Newtonian GLIDER".

STEP (3): CALCULATION OF TEMPERATURE TIME HISTORIES

For the chosen trajectory using the heat flux schedule (approxi-mated by Figure 8) the thermal response was calculated for a hypothetical arrangement of the heat shield insulation (asbestos values of thermal conductivity from Reference (11), page 382) and the structural panel. The heat capacity of the thermal structure is based on that of the structural panel (depth = .50 in.; face sheet thickness, .012 in., cell size, 3/16 in.; foil gage, .002 in.) for both the structural panel and heat shield, and the use of asbestos in this hypothetical arrangement does not imply that it could be used at such temperatures. The purpose of the above scheme is merely to introduce an appropriate thermal resistance on a realistic basis between the heat shield and structural panel so as to achieve approximately and simultaneously the maximum

TEMPERATURES DESIRED, AND SUCH THAT THE PREVIOUS TEMPERATURE VS TIME HISTORY OF EACH COMPONENT CAN BE MEANINGFUL IN TERMS OF A SPECIFIC TRAJECTORY AND THERMAL CONFIGURATION OF THE STRUCTURE. IT IS OBVIOUS THAT THERE ARE MANY POSSIBLE THERMAL PROTECTION ARRANGEMENTS WHICH COULD BE DEVISED TO ACHIEVE THE DESIRED MAXIMUM TEMPERATURES; FURTHERMORE, SLIGHT MODIFICATIONS OF THE "TYPICAL" TRAJECTORY WILL ACCOMPLISH THE SAME END.

FIGURE 9 IS A PLOT OF THE MEAN TEMPERATURE-TIME HISTORY OF THE HEAT SHIELD, THE INSULATION AND THE STRUCTURAL PANEL, CONSIDERING THE ENTIRE MASS OF EACH COMPONENT AT THE TEMPERATURES TMHS, TMI AND TMSP, RESPECTIVELY. FIGURE 10 SHOWS THE TEMPERATURE DIFFERENTIAL ACROSS THE STRUCTURAL PANEL WHICH IS COMMENSURATE WITH FIGURE 9. A SUMMARY OF THE GEOMETRIC AND THERMAL PROPERTIES OF THE MATERIALS USED TO COMPUTE THESE RESULTS IS GIVEN BELOW. (THE SAME THICKNESSES FOR THE HEAT SHIELD AS FOR THE STRUCTURAL PANEL HAVE BEEN USED FOR THE PURPOSES OF THERMAL RESPONSE ANALYSIS. THE HEAT SHIELD THERMAL RESPONSE WOULD BE SLIGHTLY FASTER FOR THE .008" FACE SHEET AND 3/8" CORE; HOWEVER, THIS DIFFERENCE COULD BE REDUCED BY A CHANGE IN THE INSULATION THICKNESS AND, THEREFORE, THE EFFECT OF THE ASSUMPTIONS LISTED BELOW ARE CONSIDERED TO INTRODUCE NEGLIGIBLE ERROR FOR THE PURPOSES INTENDED)

GEOMETRIC AND THERMAL PROPERTIES OF HEAT SHIELD, INSULATION, STRUCTURAL PANEL CONFIGURATION CB D-36

- 1. HEAT SHIELD AND STRUCTURAL PANEL
 - A. FACE SHEET: .012 IN. (= TF)
 - B. Core Thickness: .50 in. (= t_c)
 - c. Cell Size: 3/16 in. (= sc)
 - D. CORE FOIL GAGE: .002 IN. (= TCF)
 - E. MATERIAL: CB D-36 (DENSITY PG = .29 LB/IN3)
 - F. Core solid area ratio: $(A_C/A_W) = .0163$
 - G. Panel Weight/square ft.: $W/A = \left[2\tau_F + \tau_C \frac{A_C}{A_W}\right] \rho_G = 1.338 \text{ LB/ft}^2$
- Material Properties CB D-36
 - A. THERMAL CONDUCTIVITY AT 2500°F:

$$\kappa_{\text{SP}} = 42 \frac{\text{BTU}}{\text{HR.FT}^2} (\text{°F/FT}) = 1.167 \times 10^{-2} \frac{\text{BTU}}{\text{SEC.FT}^2} (\text{°F/FT})$$

(REFERENCE 14, PAGE 25)

B. Specific HEAT: C = .074 BTU LB. F

- 3. INSULATION MATERIAL ("ASBESTOS" TYPE)
 - A. THERMAL CONDUCTIVITY:

$$\kappa_1 = .23 \frac{BTU}{HR.FT^2} (°F/FT) = .64 \times 10^{-4} \frac{BTU}{SEC.FT^2} (°F/FT)$$

(EXTRAPOLATED FROM DATA OF REFERENCE 8, PAGE 382)

- B. DENSITY: $G_{P_1} = 29.3 \text{ LB.}/\text{FT}^3$
- c. Specific Heat: C₁ = .15 Btu
 LB.°F (ESTIMATE)

BASIS: TYPICAL RANGE

METALS: .03 < C < .1 BTU LB. F

GASES: .2 < C < .4

Liquips: .2 < C < 1.0

(Asbestos is considered a silica and air mixture)

D. INSULATION RESISTANCE PARAMETER:

$$R_1 = \frac{\Delta z_1}{2\kappa_1} = 43.8^{\circ} \frac{F}{BTU/SEC. FT^2}$$

E. INSULATION THICKNESS: $\Delta z_1 = .0562$ FT. = .675 IN.

The method of calculating thermal response is given in Appendix B. From Figure 9 it is determined that a maximum temperature of about 3500°R (3040°F) is achieved for the heat shield panel; how-ever, the estimated heat flux vs time schedule of Figure 8 produces a structural panel average temperature of about 2760°R (2300°F), somewhat below the target, but close enough to regard the history as having a realistic basis in a re-entry situation. Therefore, further adjustments of the heat flux (via the trajectory) or heat insulation scheme are not considered necessary.

From Figure 10 the maximum temperature gradient across the panel is found to occur early in the trajectory when the upper surface has responded, but little heat has been transferred to the lower surface by radiation (the dominant mode at high temperatures) due to the low early temperatures, and the ineffectiveness of the low solidity core (Ac/Aw = .016) in transferring heat by conduction.

2. THERMAL RESPONSE OF STRUCTURAL PANEL UNDER LABORATORY SIMULATION TIONS (STEP 4)

FIGURE 11 DISPLAYS THE ESTIMATED THERMAL RESPONSE OF THE STRUCTURAL PANEL WHEN EXPOSED TO A QUARTZ LAMP RADIANT HEAT FLUX SCHEDULE DEPICTED IN FIGURE 12B, IN WHAT IS DESIGNATED AS A "SLOW" HEATING SCHEDULE (DESIGNED TO HOLD

THE DIFFERENTIAL TEMPERATURE BETWEEN THE UPPER AND LOWER SURFACE TO 150°F. THE TEMPERATURE DIFFERENTIAL UNDER THESE CONDITIONS IS SHOWN IN FIGURE 12a). FIGURE 14 SHOWS THE TEMPERATURE DIFFERENTIAL ACROSS THE PANEL TO A "RAPID" HEATING SCHEDULE, WHERE THE TIME RATE OF CHANGE OF HEAT FLUX IS .35 BTU/FT.2 SEC.2 OR 2.57 WATTS/IN.2 PER SECOND FOR THE FIRST 100 SECONDS. THIS ATTENDANT TEMPERATURE DIFFERENTIAL REACHES A PEAK OF ABOUT 400°F. AT T = 20 SEC. FIGURE 15 SHOWS A COMPARISON OF THE FLIGHT RE-ENTRY RESPONSE AND THE POSSIBLE LABORATORY "SLOW" AND "RAPID" HEATING SCHEDULES. ALSO SHOWN ARE REFERENCE TEMPERATURE VS TIME RATES FROM 10°F/SEC. TO 40°F/SEC. THESE CURVES ARE BASED ON THE FOLLOWING ASSUMPTIONS:

- (1) THE BACK SIDE IS A PERFECT RADIATION REFLECTOR.
- (2) The heat losses are by free convection from both sides of a vertical plate. For a vertical plate of length greater than one foot, Reference 8, page 242, gives the following relation for the heat transmission loss coefficient, $H_{\rm C}$, to the air:

$$\frac{H_{c}L}{\kappa_{A}} = 0.548 \left[\left(\frac{\mu_{c}}{\kappa} \right)_{A} \left(\frac{L^{3} \rho_{A} 2 (T_{W} - T_{A})}{\mu_{A} 2 T_{A}} \right) \right] 1/4$$
(2.1)

WHERE "A" DENOTES PROPERTIES OF AIR AT AMBIENT TEMPERATURE

μ = VISCOSITY OF AIR, LB SEC/FT²

K = THERMAL CONDUCTIVITY OF AIR BTU/HR FT2 (°F/FT)

L = HEIGHT OF VERTICAL PLATE, FT.

 ρ = DENSITY OF AMBIENT AIR, SLUGS FT³

 T_{Δ} = AMBIENT AIR TEMPERATURE °R

Tw = PANEL SURFACE TEMPERATURE "R

AND

$$H_{C} = \dot{Q}_{CA}/\left(T_{W}-T_{A}\right) \tag{2.2}$$

where (Reference 8, page 242)

CA = CONVECTIVE HEAT LOSS RATE BTU/HR FT2

H_C = CONVECTIVE HEAT TRANSFER COEFFICIENT BTU/HR FT² °F

FOR AIR AT LOW TEMPERATURES, EQUATION (2.1) IS APPROXIMATED BY

$$H_C = 0.27 (\Delta T)^{0.25}; L > 1 \text{ FT.}$$
 (2.3)

(3) Half of the thermal mass of the core is lumped with each face sheet and all the remaining assumptions of Appendix B.

In examining Figures 11 to 15 observe that the maximum heat flux rate assumed is 35 Btu/sec ft² = 257 watts/in.². The Laboratory capability is estimated to be 200 watts/sq. in. for short time (possibly 5 to 10 minutes). In Figure 8 the re-entry simulation requires maintenance of maximum heat flux for only about 20 seconds, and therefore the 50% over-load should be tolerable for that period.

3. ANALYTIC STUDY OF ULTIMATE COMPRESSIVE FAILURE LOADS

Utilizing the analysis presented in Appendix C, the calculations to determine the ultimate compressive failure load for the 24" x 36" structural panels were made. Figures 16 and 17 show the results of this analytic study and emphasize the dependency of the ultimate load on the temperature differential between the upper and lower faces of the refractory honeycomb. For these cases the full panel size was used in the equations of Appendix C.

FOR BOTH THE TZM MOLYBDENUM AND THE D-36 COLUMBIUM PANELS THE SAME TRENDS EXIST. INITIALLY THE ULTIMATE COMPRESSIVE FAILURE LOAD INCREASES WITH INCREASING TEMPERATURE DIFFERENTIAL. THIS IS DUE TO THE MOVEMENT OF THE LOAD LINE TOWARD THE LOWER SURFACE OR COLDER SIDE WITH INCREASING THERMAL ECCENTRICITY. THE INCREASE IN STRENGTH ALLOWABLE WITH DECREASE IN TEMPERATURE ALLOWS FOR THE GREATER LOAD. AS THE THERMAL ECCENTRICITY INCREASES, A POINT IS REACHED WHERE THE LOWER FACE SHEET IS NO LONGER THE CRITICAL MEMBER. AT THIS POINT, FAILURE OF THE UPPER SHEET IN TENSION DETERMINES THE ALLOWABLE LOAD AND AN INCREASE IN CURVATURE PAST THIS POINT CAUSES A DECREASE IN THE ALLOWABLE COMPRESSIVE LOAD.

ALSO SEEN IN FIGURE 16 IS THE CHANGE IN ALLOWABLE COMPRESSIVE LOAD WITH A DECREASE IN THE THICKNESS OF THE CORE. IN GENERAL, THE ALLOWABLE DECREASES WITH THICKNESS OF CORE. IN ADDITION, THE SHIFT FROM FAILURE IN COMPRESSION OF THE LOWER SHEET TO FAILURE IN TENSION OF THE UPPER SHEET OCCURS AT A SMALLER TEMPERATURE DIFFERENTIAL AS THE CORE THICKNESS DECREASES. THIS OCCURS SINCE THE THERMAL ECCENTRICITY NECESSARY TO SHIFT THE LOAD LINE OUTSIDE THE SECTION DECREASES WITH DECREASING CORE THICKNESS.

For the panels being built, i.e., as suggested by ASD in the work statement, the ultimate compressive load allowed in the structure will be that as calculated for no temperature differential between upper and lower surfaces, provided the temperature differential at the panel center never exceeds 400°F for the D-36 and 500°F for the TZM.

4. PANEL DESIGN

During this period engineering drawings were prepared, establishing the design of the planned honeycomb panels. Preliminary discussion of these designs was accomplished early in December and, upon completion of the drawings, they were submitted for ASD approval. A policy decision was required in order to proceed with the designs because the engineering investigation indicated that panels constructed to the dimensions and skin gages established in the statement of work would not be of optimum structural design and would, therefore, fail at a loading condition below the target

FIGURES AS DEFINED IN THE STATEMENT OF WORK. OPTIMUM DESIGNS WERE EVOLVED FOR THE PRESCRIBED LOADING CONDITIONS; HOWEVER, THESE DESIGNS WOULD NECESSARILY DEVIATE CONSIDERABLY FROM THE PRESCRIBED PANEL THICKNESS AND SKIN GAGES. ADOPTION OF AN OPTIMUM DESIGN WOULD HAVE RESULTED IN A CONSIDERABLE INCREASE IN MATERIALS COST.

In conference with ASD Project Engineer, B. E. Price, it was established that the panel design would conform with originally established panel thicknesses and skin gages given in the statement of work. The data deRIVED FROM THE TESTING PROGRAM WILL BE ANALYZED TO ESTABLISH PREDICTED PERFORMANCE OF AN OPTIMUM DESIGN FOR EACH MATERIAL AND PANEL CONFIGURATION.

THE PANEL DESIGNS INCORPORATE CERTAIN OBJECTIVES INTENDED TO PRODUCE THE MOST USABLE STRUCTURES.

- 1. For both alloys, D-36 and TZM, the honeycomb core material is required to be diffusion bonded by a process which involves no temperatures above the recrystallization point for the material. The resulting core retains maximum ductility and toughness.
- 2. The heat shield panel design achieves a simple telescope fit between the inner and outer skins for purposes of accomplishing edge seal by brazing. The design, however, retains the desirable underlap overlap arrangement which can be oriented favorably to the air flow direction.
- 3. THE SUPPORTING CLIP DETAILS FOR THE HEAT SHIELD PANELS ARE DESIGNED FOR MACHINING SO THAT THE BRAZED JOINTS ARE HELD TO A MINIMUM AND SO THAT NO WELDING IS REQUIRED IN THE DETAIL.
- 4. Since welding of the columbium and the associated recrystallization are not harmful to the properties of the D-36 alloy, these structural panels utilize welding where advantageous.
- 5. Because of the difficulties associated with welding of molybdenum alloys, the TZM structural panels are designed with corner reinforcement pieces to be brazed in the assembly. This makes it possible to produce a brazed structural panel in molybdenum alloy without recrystallization effects in any portion of the structure.

SUCCESSFUL PRODUCTION AND TESTING OF THESE PANEL DESIGNS IS CONTINGENT ON THE ABILITY TO PROTECT BRAZED JOINTS FROM THE HIGH TEMPERATURE OXIDATION EFFECTS AS WELL AS PARENT METAL. THIS REQUIREMENT IS BEING INVESTIGATED. COMPATIBILITY OF PROTECTIVE COATINGS AND THE SELECTED BRAZE ALLOYS IN TYPICAL JOINTS IS PLANNED FOR TESTING.

C. BRAZING STUDY

1. PRE-BRAZE CLEANING PROCEDURES

TZM CLEANING

During this reporting period concentrated effort was placed upon development of cleaning procedures for the TZM molybdenum alloy. The cleaning procedure

EVALUATION WAS CONDUCTED ON 2" x 1" x .010" SPECIMENS. THESE SPECIMENS WERE WEIGHED BEFORE AND AFTER THE ETCHING STEP OF EACH CLEANING PROCEDURE TO DETERMINE PER CENT WEIGHT LOSS.

THE CLEANING PROCEDURES INVESTIGATED AND THE RESULTS OBTAINED ARE RECORDED IN TABLE 1. Some of the specimens were pre-oxidized five minutes at 800°F IN AIR BEFORE CLEANING IN ORDER TO SIMULATE THE OXIDATION THAT COULD OCCUR FROM HEAT GENERATED IN CORE MACHINING OPERATIONS. CLEANING PROCEDURES 1 AND 5 WERE EXCESSIVELY REACTIVE AND ALSO STAINED THE SPECIMENS. CLEANING PROCEDURES 4, 6, 2, AND 3 DID NOT PRODUCE APPRECIABLE CLEANING AS EVIDENCED BY LOW SPECIMEN WEIGHT LOSS AND INABILITY OF THE CLEANING PROCEDURES TO REMOVE THE BLUISH OXIDE ON THE PRE-OXIDIZED SPECIMENS.

CLEANING PROCEDURE 7 READILY REMOVED THE SURFACE OXIDE ON THE PRE-OXIDIZED SPECIMENS AND WAS FOUND TO PRODUCE EXCELLENT RESULTS ON THE TZM SHEET SPECIMENS. A TZM HONEYCOMB SANDWICH SPECIMEN, UTILIZING CLEANING PROCEDURE 7, WAS BRAZED ONE (1) MINUTE AT 2875°F IN A 300 MM. ARGON ATMOSPHERE WITH THE TI-13V-11CR-3AL BRAZE ALLOY. EXCELLENT BRAZE ALLOY FILLETING AND NODE FLOW WAS OBSERVED. THIS CLEANING PROCEDURE OFFERS EXCELLENT POTENTIAL FOR PRE-BRAZE CLEANING OF TZM ALLOY AND IT WILL BE EVALUATED FURTHER UNDER MANUFACTURING CONDITIONS.

THE SPECIFIC STEPS INVOLVED IN THE TZM CLEANING PROCEDURE ARE DETAILED IN TABLE 2.

D-36 CLEANING

Several samples of D-36 honeycomb core (3/16" cell size x .002" foil) with a heavy visible surface contamination were cleaned five (5) minutes at room temperature in the 2HF-25HNO₃-73H₂O cleaning procedure discussed in the previous progress report. These samples were diffusion bonded with no intermediate metal. Weight loss data obtained on 1" x 1" x .5" core samples follows:

SAMPLE	INITIAL WEIGHT	FINAL WEIGHT	% WT. Loss
1	1.4507 gm.	1.4327 gm.	1.24
2	1.5205	1.5002	1.34
3	1.5732	1.5535	1.25

Visual examination of these samples showed excellent cleaning. In addition, one of the samples was used for brazing a honeycomb sandwich specimen using face sheets which were similarly cleaned. The specimen was brazed one (1) minute at 2900°F in a 300 mm. Argon atmosphere using the Ti-13V-11Cr-3AL braze alloy. Excellent filleting and node flow was observed. This cleaning procedure offers excellent potential for pre-braze cleaning of D-36 alloy and it will be evaluated further under manufacturing conditions.

THE SPECIFIC STEPS INVOLVED IN THE D-36 CLEANING PROCEDURE ARE DETAILED IN TABLE 2.

BRAZE ALLOY CLEANING

THE D-36 CLEANING PROCEDURE OUTLINED IN TABLE 2 WAS EVALUATED AS A CLEANING PROCEDURE FOR THE PURE TITANIUM AND TI-13V-11CR-3AL BRAZE ALLOYS. EXCELLENT

RESULTS WERE OBTAINED WITH IMMERSION TIMES OF 1-3 MINUTES IN THE ACID ETCH. WEIGHT LOSS ON .002" TITANIUM AND TI-13V-11CR-3AL FOILS RANGED FROM 5-20% FOR THE 1-3 MINUTE IMMERSION TIMES IN THE ACID ETCH. THIS CLEANING PROCEDURE WILL BE INVESTIGATED UNDER MANUFACTURING CONDITIONS.

2. BRAZE ALLOY SELECTION AND EVALUATION

A number of "T" Joint Specimens, of the type described in the previous progress report, and small honeycomb sandwich specimens were brazed to establish the brazing temperature ranges for the Ti-13V-11Cr-3AL and pure titanium braze alloys on D-36 and TZM. The specimens and braze alloys were chemically cleaned according to the procedures outlined in Table 2. Brazing was performed in a 300 mm. Argon atmosphere using a heating rate of 650°F/min. And holding one minute at the braze temperature. Determination of the optimum brazing temperature range was based on braze alloy filleting, flow, and erosion behavior.

THE OPTIMUM BRAZING TEMPERATURE RANGE FOR THE TI-13V-11CR-3AL ALLOY WAS FOUND TO BE 2850-2900°F ON BOTH THE TZM AND D-36. TEMPERATURES ABOVE 2900°F PRODUCED HEAVY EROSION WHEREAS BRAZING TEMPERATURES BELOW 2850°F PRODUCED MARGINAL FILLETING AND FLOW. THE OPTIMUM BRAZING TEMPERATURE RANGE FOR THE PURE TITANIUM BRAZE ALLOY ON D-36 AND TZM WAS ESTABLISHED AT 3150-3200°F. BRAZING ABOVE OR BELOW THIS RANGE PRODUCED EFFECTS SIMILAR TO THOSE OBSERVED WITH THE TI-13V-11CR-3AL ALLOY.

For reasons discussed in the brazing envelope protection section of this report, it would be highly desirable to braze the D-36 structural panels in the 2750-2800°F range rather than a 2850-2900°F range as required for the Ti-13V-11Cr-3AL braze alloy. Therefore, two modifications of this alloy have been formulated to lower the brazing temperature range approximately 100°F. The nominal composition of these alloys is as follows: (1) Ti-20V-20Cr, (2) Ti-13V-20Cr.

The alloys are being prepared in the form of 100 gm. Arc-cast buttons which will be evaluated for applicability to brazing of the D-36 structural panels.

3. TZM RECRYSTALLIZATION STUDIES

RECRYSTALLIZATION STUDIES WERE CONDUCTED ON .011" TZM SHEET AND .002"
TZM FOIL TO AID IN ESTABLISHING PERMISSIBLE BRAZE TEMPERATURES FOR THE
TZM STRUCTURAL PANELS. THE SPECIMENS WERE HEATED APPROXIMATELY 650°F/
MIN., HELD SIX (6) MINUTES AT TEMPERATURE, AND COOLED BELOW 1500°F WITHIN ONE (1) MINUTE. ALL THERMAL EXPOSURES WERE CONDUCTED IN A VACUUM
OF 0.2 MICRONS OR BETTER. THE AMOUNT OF RECRYSTALLIZATION WAS DETERMINED BY VISUAL EXAMINATION.

THE RECRYSTALLIZATION RESULTS ARE RECORDED IN TABLE 3 AND PLOTTED IN FIGURE 18. THE RECRYSTALLIZATION TEMPERATURE RANGE FOR THE .011" SHEET WAS APPROXIMATELY 2400-2700°F. THE RECRYSTALLIZATION TEMPERATURE RANGE FOR THE .002" FOIL WAS APPROXIMATELY 2100-2500°F. THUS, THE RECRYSTALLIZATION RANGE OF THE .002" FOIL WAS 200-300°F LOWER THAN THE RECRYSTALLIZATION RANGE FOR THE .011" SHEET MATERIAL.

REPRESENTATIVE MICROSTRUCTURES OF THE SHEET AND FOIL SPECIMENS AFTER THERMAL EXPOSURES ARE SHOWN IN FIGURES 19 AND 20. THE FOIL SPECIMENS EXPOSED TO THE ELEVATED TEMPERATURES SHOWED NO SIGNS OF DELAMINATION WHEN EXAMINED METALLOGRAPHICALLY. However, THE AS-RECEIVED SPECIMEN EXHIBITED SEVERE DELAMINATION. SEVERAL ADDITIONAL AS-RECEIVED SPECIMENS WERE MOUNTED IN BAKELITE AND A ROOM TEMPERATURE-CURING MOUNT MATERIAL. ALL OF THESE SPECIMENS EXHIBITED SEVERE DELAMINATION.

IT APPEARED THAT METALLOGRAPHIC PREPARATION CAUSED THE DELAMINATION TO FORM IN THE AS-RECEIVED FOIL. However, THERMAL EXPOSURES APPARENTLY REDUCED THE DELAMINATION TENDENCY OF THE FOIL. AT THE PRESENT TIME, IT IS NOT CLEAR WHETHER THE REDUCTION IN DELAMINATION TENDENCY WAS DUE TO STRESS RELIEF AND/OR OTHER MECHANISMS.

Based on the above data, it is recommended that all TZM foil be stressrelieved after rolling and prior to any core manufacturing operations.

SEVERAL SLIT STRIPS OF .002" FOIL FROM ANOTHER HEAT OF TZM FOIL WERE STRESS RELIEVED FOUR (4) MINUTES AT 2000°F AND SENT TO HEXCEL PRODUCTS, INC. FOR FABRICATION OF A DIFFUSION BONDED HONEYCOMB SPECIMEN. THIS SAMPLE REPORTEDLY DELAMINATED DURING THE HOBE EXPANSION OPERATION. THUS, THE STRESS-RELIEF MAY HAVE ALLEVIATED BUT DID NOT ELIMINATE THE DELAMINATION PRODUCED IN THE SEVERE TEST REPRESENTED BY THE HOBE EXPANSION. ADDITIONAL THERMAL EXPOSURES WILL BE STUDIED TO DETERMINE THE EFFECTS OF VARIOUS TIMES AND TEMPERATURES ON DELAMINATION DURING CORE FABRICATION.

If the TZM panels are to be brazed with no recrystallization, Figure 18 indicates a maximum braze temperature of about 2100°F based on recrystallization behavior of the foil. This would limit service temperatures considerably.

WHILE IT IS GENERALLY BELIEVED THAT RECRYSTALLIZATION EMBRITTLES MOLYBDENUM ALLOYS, RECENT IN-HOUSE WORK AT NORTHROP-NORAIR (REFERENCE 3) INDICATED THAT THE TM AND TZM ALLOYS WERE NOT SUBSTANTIALLY EMBRITTLED BY RECRYSTALLIZATION PER SE. HOWEVER, THE GRAIN GROWTH OCCURING AFTER RECRYSTALLIZATION SEVERELY EMBRITTLED THE TM AND TZM. IF ONE MAY ACCEPT THE LOSS IN STRENGTH CAUSED BY RECRYSTALLIZATION, APPRECIABLY HIGHER BRAZING TEMPERATURES MAY BE TOLERATED WITH NO SUBSTANTIAL EMBRITTLEMENT.

FIGURE 18 SHOWS THAT A BRAZE TEMPERATURE OF 2400°F WILL PRODUCE 40% RE-CRYSTALLIZATION OF THE .002" FOIL AND ABOUT 2% RECRYSTALLIZATION OF THE .011" SHEET. THEREFORE, ATTEMPTS WILL BE MADE TO BRAZE THE TZM STRUCTURAL PANELS AT APPROXIMATELY 2400°F WHICH WILL PRODUCE SOME RECRYSTALLIZATION AND LOSS OF STRENGTH, BUT NO SUBSTANTIAL EMBRITTLEMENT.

4. Brazing Envelope Protection

CONVENTIONAL COATING APPLICATION METHODS WERE NOT CONSIDERED APPLICABLE TO ENVELOPE COATING FOR REASONS DISCUSSED IN THE FIRST QUARTERLY PROGRESS REPORT ON THIS CONTRACT. THEREFORE, DURING THIS REPORTING PERIOD, A NUMBER OF FLAME SPRAYED PROTECTIVE COATINGS WERE EVALUATED ON PURE TITANIUM AND THE CB-33TA-.7ZR(F82) ALLOY. THESE COATING TESTS WERE CONDUCTED BY RESISTANCE HEATING COATED SPECIMENS AS DESCRIBED IN THE PREVIOUS PROGRESS REPORT. THE TEST RESULTS ARE RECORDED IN TABLE 4.

TITANIUM PROTECTION

Specimens TA, TB, and TC were uncoated samples tested in air at 2400, 2600, and 2900°F, respectively, to establish a base line for comparison. Specimens TD, TE, and TF were uncoated samples run in argon to determine whether high temperatures per se would embrittle the titanium. These samples exhibited extreme grain growth and an "orange peel." surface but they were ductile at room temperature. Figure 21 shows the microstructures of the as-received titanium and the sample heated two (2) minutes at 2900°F in argon. The extreme grain growth is evident. In addition, the thermally exposed sample retained the beta structure.

ALL OF THE .012" TITANIUM SAMPLES COATED WITH AL203-2.5TI02 AND ALUMINUM SEALER COMBINATIONS WITHSTOOD TEN (10) MINUTES EXPOSURE AT 2400°F AND 2600°F. However, these specimens were severely embrittled as determined by bending the samples and by microhardness measurements. Figure 22 shows the microstructures of specimens T11 and T12 exposed ten (10) minutes at 2400°F and 2600°F, respectively. Specimen T11 showed a retained alpha structure at the surface, probably resulting from the alpha stabilizing effect of oxygen. The center exhibited a beta structure. The microhardness data showed a large increase in hardness at the surface as well as the center. The microstructure of specimen T12 showed similar results except that isolated alpha grains extended well below the surface, suggesting greater oxygen penetration.

Specimen Life at 2900°F ranged from 1.1-5.9 minutes. Considerable scatter was noted in the failure times at 2900°F. In general, it was difficult to correlate the effects of coating thickness combinations on specimen Life from the data obtained.

In view of the extreme embrittlement and hardness increase observed in the coated specimens, specimen TG was exposed two (2) minutes at 2400°F in argon to determine the effect of the AL203-2.5TiO2 on the titanium in the absence of air. This specimen was highly embrittled indicating an attack of the substrate by the coating. Figure 23 shows the microstructure of this specimen. The titanium exhibited a completely alpha structure, high hardness, and severe cracking along the interface. Specimen TH, coated with ZrO2 and specimens TJ through TN, coated with proprietary frits, were thermally exposed in a similar manner. The results paralleled those obtained on specimen TG.

One set of .032" thick specimens (T24 and T25) were tested with the AL₂0₃-2.5Ti0₂ coating with aluminum sealer to determine if a thicker substrate would minimize the embrittlement caused by the coating attack. However, the results closely paralleled those obtained on the .012" thick material.

IT APPEARS THAT CERAMIC COATINGS IN GENERAL ATTACK AND EMBRITTLE
TITANIUM AT THE THERMAL EXPOSURES OF INTEREST. WHILE IT IS WELL KNOWN
THAT MOLTEN TITANIUM WILL ATTACK CERAMICS, IT WAS RATHER SURPRISING
THAT THE TITANIUM WAS RAPIDLY ATTACKED IN THE SOLID STATE AT SUBSTANTIALLY LOWER TEMPERATURES SINCE FREE ENERGY DATA INDICATES OTHERWISE.

On the basis of these results, ceramic coatings were not considered further and effort shifted to metallic coatings for oxidation protection.

Specimen T22 was a .012" thick titanium sample coated with chromium and aluminum. This sample failed in a relatively short time at 2900°F due to incipient melting. It was believed that the aluminum and particularly the chromium diffused into the titanium to depress the melting point below 2900°F. Therefore, the .012" titanium was replaced with .032" titanium in subsequent tests in order to provide a greater "diffusion sink".

SPECIMENS T26 THROUGH T35 WERE COATED WITH COMBINATIONS OF PURE CHROMIUM AND PURE ALUMINUM. THESE COATINGS PROTECTED THE TITANIUM RATHER WELL AT 2400 AND 2600°F. THE HARDNESS INCREASE ON SAMPLES AFTER TESTING WAS RELATIVELY LOW INDICATING MILD EMBRITTLEMENT. AT 2900°F, SPECIMEN LIFE RANGED FROM .1-3.75 MINUTES FOR THE VARIOUS COATING COMBINATIONS. AGAIN IT WAS DIFFICULT TO CORRELATE THE EFFECTS OF COATING COMBINATIONS ON SPECIMEN LIFE.

FIGURE 24 SHOWS THE MICROSTRUCTURES OF SPECIMENS T28 AND T26 EXPOSED TEN (10) MINUTES AT 2400°F AND 2600°F RESPECTIVELY. A HEAVY DIFFUSION ZONE BETWEEN THE COATING AND SUBSTRATE WAS OBSERVED. IN ADDITION, THE COATING LAYER CRACKED WHEN THE SPECIMEN COOLED BELOW 1000°F UNDER THE RESTRAINT IMPOSED BY THE RESISTANCE HEATING CLAMPS. HOWEVER, THE SUBSTRATE WAS SUFFICIENTLY DUCTILE TO PREVENT THE SURFACE CRACKS FROM PROPAGATING. THE SURFACE CRACKS WERE BELIEVED TO HAVE RESULTED FROM EMBRITTLEMENT OF THE CHROMIUM BY NITROGEN ABSORPTION DURING THE TEST. THIS TYPE OF COATING APPEARS VERY PROMISING FOR PROTECTION OF THE TITANIUM BRAZING ENVELOPES TO AT LEAST 2600°F. However, AT 2900°F THE COATING BEHAVIOR WAS ERRATIC. THE SPECIMENS COATED WITH MOSI2 APPEARED TO OFFER GOOD PROTECTION AT 2600°F AND EXHIBITED PROMISE AT 2900°F. However, THE RESULTS AT 2900°F AGAIN SHOWED CONSIDERABLE SCATTER. THIS COATING WILL BE EVALUATED FURTHER.

CB-33TA-. 7ZR PROTECTION

Specimens C1 and C2 were coated with AL203-2.5Ti2 and heated two (2) minutes in argon at 2900°F and 3200°F, respectively. Specimens C3 and C4 were coated with ZrO2 and similarly treated. All of these specimens were ductile after thermal exposure. Thus, these coatings are compatible with the columbium alloy whereas they severely attacked the pure titanium.

Specimens C5, 6, 7, 13, 14, 15 and 16 were coated with the AL2O3-2.5TIO2 and Aluminum sealer combinations. Specimens C5, 6, and 7 showed some promise at 2900°F and 3200°F. Specimens C13 and C14 which were coated with AL2O3-2.5TIO2 and no aluminum sealer showed poorer life at 3200°F than similar specimens including the sealer. Thus, the aluminum sealer appeared to reduce coating porosity to some extent. However, all of these specimens showed excessive oxygen penetration through the coating, indicating the need for more effective reduction in coating porosity.

Specimens C8, 9, and 10 were coated with pure chromium and pure aluminum. Specimen C8 showed promise at 2900° F but specimens C9 and 10, tested at 3200° F, exhibited somewhat poorer protection than the $Al_2O_3-2.5TiO_2$ and aluminum coatings.

Specimens C11 and C12 were coated with $MoSi_2$ and showed poor life at 2900^{0} F. Metallographic analysis of these specimens is in progress to determine the reasons for the poor oxidation protection of this coating.

GENERAL COMMENTS

Work to date has indicated that the maximum temperature that can be sustained by the titanium is $2900^{\circ}F_{\bullet}$. This is due in part to the melting point.

Thus, it does not appear feasible to braze the D-36 structural panels with the Ti-13V-11Cr-3Al braze alloy in titanium envelopes. This results from the fact that the brazing range is 2850-2900°F which would result in localized areas of the envelope reaching temperatures as much as 100°F above the braze temperature. An approach to this problem which will still permit use of titanium envelopes is discussed in the braze alloy selection and evaluation section of this report.

THE RESISTANCE HEATING METHOD USED TO OBTAIN THE DATA TO DATE WAS SUBJECT TO SOME SERIOUS PROBLEMS. THE RESISTANCE HEATING METHOD WAS HIGHLY DEPENDENT UPON SPECIMEN GEOMETRY. THUS, LOCALIZED COATING FAILURES CAUSED THERMAL GRADIENTS, ACCELERATED FAILURES, AND DIFFICULTIES IN CORRELATING RESULTS. THIS PROBLEM WAS PARTICULARLY ACUTE IN THE TITANIUM SAMPLES TESTED AT 2900°F WHICH WAS LESS THAN 150°F BELOW THE MELTING POINT OF TITANIUM. IN ADDITION, THE DIFFERENCE IN ELECTRICAL PROPERTIES OF THE COATING AND SUBSTRATE APPEARED TO CAUSE THERMAL GRADIENTS ACROSS THE THICKNESS OF THE SPECIMENS.

TO ALLEVIATE THESE PROBLEMS, THE RESISTANCE HEATING METHOD IS BEING REPLACED WITH QUARTZ LAMP HEATING. THERMOCOUPLES WILL BE ATTACHED TO THE SPECIMENS FOR TEMPERATURE MEASUREMENT AND CONTROL. THIS WILL ALSO PERMIT A STUDY OF THERMOCOUPLE ATTACHMENT METHODS AND THERMOCOUPLE COMPATIBILITY WITH THE COATINGS.

D. MANUFACTURING PROCESS DEVELOPMENT

1. HONEYCOMB CORE FABRICATION

During this reporting period different methods and materials for formation of node bonds were studied. With the columbium D-36 alloy, both the stop-weld system and the interface metal system were systematically evaluated.

STOP-WELD SYSTEM

REAGENT GRADE MAGNESIUM OXIDE WAS FOUND TO BE SATISFACTORY FOR USE AS A STOP-OFF WITH D-36 ALLOY. THERE WAS NO REACTION OBSERVED BETWEEN THE MGO AND THE D-36 IN VACUUM FOR TEMPERATURES UP TO 2200°F. IT WAS NOTED, HOWEVER, THAT U.S.P. GRADE MGO, IF USED AS A STOP-OFF, CAUSES DISCOLORATION AND SURFACE CONTAMINATION OF THE D-36. THE BONDING CONDITION TO OBTAIN A STRONG NODE BOND FOR D-36 WITH THE REAGENT GRADE MGO AS A STOP-OFF WAS DETERMINED TO BE 2000°F FOR 10 MINUTES.

INTERFACE METAL SYSTEM - D-36

Both titanium and zirconium have been evaluated as intermediate metals with the D-36 alloy. The bonding conditions were 1400°F to 1500°F for 10 minutes. Titanium and zirconium seem to work equally well as intermediate materials. Titanium was employed in the form of foil (.001"), titanium hydride powder, or as a thin film (3 - 240 micro-inches). The zirconium was employed as foil or as a hydride powder. The use of foil as an interface material presents a problem of melting temperature limitation in the subsequent brazing of the honeycomb panels. There is also a problem of compatibility between the interface metal and with the solutions required to clean the honeycomb preparatory to the brazing operation. These problems also apply in the case of the titanium and zirconium hydride. The use of hydrides presents an additional problem of placing the material in the exact locations desired.

SELECTION OF A THIN FILM OF TITANIUM AS THE INTERFACE METAL EFFECTIVELY ELIMINATES THE PROBLEMS OF MELTING TEMPERATURE AND CLEANING COMPATIBILITY ASSOCIATED WITH OTHER AVENUES OF APPROACH. THE SELECTION OF TITANIUM FILM FOR EVALUATION STUDIES INCLUDED THOSE HAVING THICKNESSES IN 3, 5, 10, 30, 60, AND 240 MICRO-INCHES. THE THREE HEAVIEST FILMS, NAMELY 30, 60 AND 240 MICRO-INCHES, PRODUCED STRONG BONDS. FIGURE 25 SHOWS A TYPICAL D-36 BOND MADE WITH A 60 MICRO-INCH FILM OF TITANIUM.

INTERFACE METAL SYSTEM - TZM

THE INTERFACE METAL METHOD HAS BEEN SELECTED FOR FABRICATION OF THE LARGE PANELS SINCE THE TEMPERATURE REQUIRED TO OBTAIN A SATISFACTORY BOND BETWEEN RIBBONS OF TZM WITHOUT THE USE OF AN INTERFACE METAL IS ABOVE THE TZM RECRYSTALLIZATION TEMPERATURE. RESULTS OBTAINED HAVE DEMONSTRATED THAT TITANIUM FORMS A STRONG BOND WITH TZM WITH A BONDING CYCLE OF 10 MINUTES AT 1600°F. TITANIUM FOIL (.001"), TITANIUM HYDRIDE AND TITANIUM IN FILM THICKNESSES HAVE BEEN EVALUATED. THE USE OF TITANIUM FOIL PRESENTS THE SAME DISADVANTAGES WITH TZM AS WITH THE D-36 DISCUSSED ABOVE. THE USE OF

TITANIUM HYDRIDE PERMITS THE FORMATION OF A STRONG BOND WITH THE TZM. AS AN INTERESTING SIDELIGHT, IT WAS NOTED THAT THE TZM BONDED WITH THE HYDRIDE IS LESS MICACEOUS THAN THAT JOINED USING OTHER METHODS (FIGURE 26). However, This method requires approximately .002" thickness of titanium to form an adequate bond. This circumstance therefore, creates the same problems as the .001" foil. Titanium film in thicknesses of 3 micro-inches to 240 micro-inches was evaluated. It was observed that with a titanium thickness of 30 micro-inches or greater, a sound bond is formed. The titanium in the 30 to 40 micro-inch thickness provides a particularly sound bond and appears to effectively eliminate the unalloyed titanium bond in the Joint region when foil thicknesses are employed.

COLUBMIUM ALLOY (D-36), COLUMBIUM - 1% ZIRCONIUM ALLOY AND VANADIUM FOILS WERE ALSO EVALUATED AS POSSIBLE CHOICES FOR AN INTERFACE METAL TO BE USED IN CONJUNCTION WITH THE TZM. THE BONDS OBTAINED WITH TEMPERATURES TO 2200°F WERE INADEQUATE IN STRENGTH TO PERMIT EXPANSION OF THE HONEYCOMB. HIGHER TEMPERATURES WERE NOT EVALUATED BECAUSE OF CONCOMITANT RECRYSTALLIZATION OF THE TZM.

HOBE FABRICATION

THE TWO METHODS CONSIDERED FOR HOBE FABRICATION ARE THE STOP-WELD METHOD AND THE INTERFACE METAL METHOD. THE DECISION HAS BEEN MADE IN FAVOR OF THE INTERFACE METAL TECHNIQUE BECAUSE OF THE FOLLOWING REASONS:

- (1) THE SELECTION OF A NON-CONTAMINATING STOP-WELD MATERIAL AND ASSOCIATED PROBLEMS OF PLACEMENT AND REMOVAL ARE PRECLUDED.
- (2) The bonding temperatures are substantially lower with the interface metal approach (1500°F vs 2000°F).
- (3) AN INTERFACE METAL TECHNIQUE IS ESSENTIAL FOR THE TZM MOLYBDENUM ALLOY TO AVOID RECRYSTALLIZATION.
- (4) SELECTION OF THIS PROCESS OFFERS OPPORTUNITY FOR FABRICATION OF BOTH ALLOYS WITH THE SAME PROCESS.
- (5) Successful bonding utilizing interface metals of micro-inch film thickness and subsequent Thorough diffusion of this metal precludes the Limiting remelt temperature problems.
- (6) THERE IS AN INDICATED BENEFICIAL PRESSURE DISTRIBUTION EFFECT WITH THE USE OF AN INTERFACE METAL. THIS HOWEVER, HAS YET TO BE STUDIED ON THE LARGE PANEL FABRICATION PHASE OF THE WORK.

HOBE EXPANSION

COLUMBIUM FOIL IN FULL HARD CONDITION HAS CAUSED SOME DIFFICULTY IN ATTEMPTS TO OBTAIN FULL EXPANSION CONFIGURATION. HOWEVER, EXCELLENT NODE INTEGRITY HAS BEEN REALIZED WHICH WILL ASSIST IN MAKING FULL EXPANSION FEASIBLE. AS AN ALTERNATE PROCEDURE, THE FOIL CAN BE VACUUM ANNEALED TO FACILITATE EXPANSION.

A MAJOR PROBLEM IN EXPANDING TZM STEMS FROM THE MICACEOUS BEHAVIOR OF TZM. MICACEOUS BEHAVIOR MUST BE ELIMINATED BY PROPER PROCESSING OF THE FOIL BY THE SUPPLIER OR BY SUITABLE SUBSEQUENT PROCESSING TECHNIQUES. METHODS TO BE CONSIDERED ARE:

- (1) STRESS RELIEVING IN VACUUM.
- (2) STRESS RELIEVING IN HYDROGEN.
- (3) REINFORCED NODES.
- (4) APPLICATION OF TRACE AMOUNTS OF TITANIUM HYDRIDE.

ELEVATED TEMPERATURE (300 - 400°F) EXPANSION OF THE TZM HAS BEEN EVALUATED. THIS DID NOT CORRECT THE MICACEOUS BEHAVIOR OF THE TZM BUT DID RESULT IN A REDUCTION OF 45° CRACKING.

SATISFACTORY SURFACING IN THE HONEYCOMB WHILE IN HOBE FORM HAS BEEN ACCOMPLISHED BY MILLING, USING A CARBIDE CUTTING HEAD. THE CUTTING HEAD USED
IS A WENDT SONIS, STYLE 700, 4 FLUTE, 1/2" SOLID CARBIDE CUTTER. THE CUTTER
SPEED IS 560 FT. PER MINUTE WITH A TABLE SPEED OF ONE INCH PER MINUTE.
GRINDING HAS BEEN ATTEMPTED BUT PROVED UNSATISFACTORY BECAUSE OF THE EXCESSIVE HEAT GENERATED BY THIS TECHNIQUE. BELT SANDING HAS YET TO BE
EVALUATED.

2. TZM FORMING TESTS

THE TZM STRUCTURAL PANEL DESIGN INCLUDES A CORNER REINFORCEMENT CLIP WHICH INVOLVES RATHER DIFFICULT FORMING. IN ORDER TO VERIFY THE FEASIBILITY OF THE DESIGN AND TO DEVELOP FAMILIARITY WITH THE FORMING CHARACTERISTICS OF THE MOLYBDENUM ALLOY, A SIMPLE FORMING DIE WAS FABRICATED AND TESTED IN AN ELECTRICALLY HEATED HOT PLATEN PRESS. SOME AVAILABLE .040 GAGE TZM ALLOY FROM A NORTHROP IN-HOUSE PROJECT WAS TEST FORMED IN THE FIXTURE. SUFFICIENT TESTS WERE CARRIED OUT TO DEVELOP A SUITABLE BLANK, TO ESTABLISH THE USEFULNESS OF MOLYBDENUM DISULPHIDE AS A FORMING LUBRICANT AND TO VERIFY THE PRODUCIBILITY OF THE CLIP DESIGN. WHEN TZM ALLOY IS RECEIVED IN THE CORRECT GAGE (.025) THIS TOOL WILL BE DEVELOPED FOR FABRICATION OF THE REQUIRED CORNER CLIPS. THE REPORTED TESTS WERE CARRIED OUT AT A TEMPERATURE OF 380°F FOR CONVENIENCE IN THE HOT PLATEN PRESS. THE LOWER TEMPERATURE LIMIT OF FORMABILITY WAS NOT INVESTIGATED.

FIGURE 27 SHOWS THE TEST FORM DIE USED AND PART AFTER FIRST STAGE FORMING AT APPROXIMATELY 200°F. FIGURE 28 SHOWS FORMING RESULTS DURING THE BLANK DEVELOPMENT STAGE. BLANK CONFIGURATION IS CRITICAL TO SUCCESSFUL SECOND STAGE FORMING; THE CONFIGURATION OF THE BLANK SHOWN AT TOP CENTER BEING USED FOR THE THREE UNTRIMMED PARTS AT THE BOTTOM OF THE PHOTOGRAPH. SHOWN AT TOP LEFT IS A FRACTURED BLANK, THE RESULT OF AN ATTEMPT TO FORM THE FIRST BEND AT ROOM TEMPERATURE.

3. QUARTZ LAMP BRAZING FACILITY

FABRICATION OF WATER COOLED STEEL REFLECTORS TO REPLACE THE ALUMINUM REFLECTORS IN THE TEST BRAZE FACILITY WAS COMPLETED DURING THIS REPORTING PERIOD.

THE NEW REFLECTIVE SURFACES ARE GOLD FIRED FOR MAXIMUM INFRARED REFLECTIVITY. THIS UNIT WILL PERMIT TEST BRAZING OF REFRACTORY PANELS UP TO 12"x 12" IN SIZE.

SIX ADDITIONAL IGNITRON POWER CONTROL UNITS HAVE BEEN ACQUIRED AS PART OF THE NORTHROP-FUNDED MODIFICATIONS TO THE NORTHBRAZE SYSTEM. THESE WILL RAISE THE POWER CONTROL CAPABILITY TO 2100 KVA AT 600 V CONTINUOUS TOTAL FOR 12 ZONES OF CONTROL.

REQUEST WAS MADE DURING THE FIRST QUARTER OF THIS PROGRAM FOR AUTHORIZATION TO MODIFY AND INSTALL TWO AIR FORCE PROPERTY TRANSFORMERS WHICH WOULD BRING THE TOTAL POWER SOURCE TO 2000 KVA AT 600 V CONTINUOUS RATING. AUTHORIZATION HAS BEEN WITHHELD PENDING RESULTS OF A SEARCH TO LOCATE SIMILAR SURPLUS EQUIPMENT. AT PRESENT NO ANTICIPATED APPROVAL DATE CAN BE ESTABLISHED.

ALTHOUGH THE TEST BRAZEMENTS IN PHASE I OF THE PROGRAM CAN BE ACCOMPLISHED WITH EXISTING FACILITIES, THE ORIGINAL PROGRAMMING SCHEDULED THE NEW INSTALLATION AND THE ATTENDANT REARRANGEMENT OF THE POWER CONTROL UNITS TO BE COMPLETED BEFORE THE TEST BRAZEMENT PORTION OF THIS PHASE WAS TO BEGIN.

MATERIALS AND FACILITIES WOULD THEN BE SIMULTANEOUSLY AVAILABLE FOR A CONTINUOUS PROGRAM OF BRAZING EFFORT. IT APPEARS UNAVOIDABLE THAT INTERRUPTIONS IN THE PHASE I BRAZE PROGRAM WILL OCCUR DUE TO LACK OF POWER AND/OR POWER CONTROL DURING A FUTURE TRANSFORMER INSTALLATION AND POWER CONTROLLER CHANGE-OVER.

4. CONCLUSIONS AND RECOMMENDATIONS

THE WORK PERFORMED DURING THIS PERIOD PERMITS THE FOLLOWING CONCLUSIONS AND RECOMMENDATIONS TO BE MADE:

- 1. PRE-BRAZE CLEANING PROCEDURES HAVE BEEN DEVELOPED FOR D-36, TZM AND THE TITANIUM-BASE BRAZE ALLOYS.
- 2. Based on recrystallization and embrittlement considerations, the maximum braze temperature for the TZM structural panels was determined to be approximately 2400°F.
- 3. Delamination of TZM foil is a severe obstacle to the expansion of diffusion bonded HOBE from this material. Some post rolling processes investigated show preliminary promise in alleviating this characteristic. These should be pursued. Suppliers of TZM foil should seek to isolate and eliminate the cause of this tendency to delaminate.
- 4. A 30-40 MICRO-INCH DEPOSIT OF TITANIUM PRODUCES AN EXCELLENT NODE BOND IN TZM HONEYCOMB CORE, SUITABLE FOR THE STRUCTURAL PANEL CONCEPT. 30, 60 AND 240 MICRO-INCH DEPOSITS PRODUCED SOUND BONDS IN D-36. THE THINNEST FILM WITH WHICH A SOUND BOND IN D-36 CAN BE MADE HAS NOT BEEN DETERMINED; THIS LOWER LIMIT SHOULD BE EXPLORED FOR USE ON THE D-36 HEAT SHIELD PANELS.

5. FUTURE WORK

THE FOLLOWING WORK IS SCHEDULED FOR THE NEXT QUARTER:

- 1. INITIATE THE TEST BRAZING SECTION OF PHASE 1.
- 2. Complete the design and construction of D-36 and TZM fabrication tooling.
- 3. EVALUATE TI-20V-20CR AND TI-13V-30CR BRAZE ALLOYS FOR USE ON D-36 STRUCTURAL PANELS.
- 4. EVALUATE D-36 HONEYCOMB CORE NODE BONDS WITH VARIOUS MICRO-INCH DEPOSITS OF TITANIUM AT BRAZING CYCLES 2850-3200°F.
 FINALIZE MANUFACTURING PROCESS SPECIFICATIONS FOR FABRICATION OF PRODUCTION QUANTITIES OF D-36 DIFFUSION BONDED CORE.
- 5. EVALUATE MANUFACTURING PROCESSES FOR THE FABRICATION OF DIFFUSION BONDED TZM CORE AND FINALIZE PROCESS SPECIFICATIONS FOR FABRICATION OF PRODUCTION QUANTITIES OF TZM DIFFUSION BONDED CORE.

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APPENDIX A

AERODYNAMIC HEATING AND RADIATION EQUILIBRIUM CALCULATIONS

APPENDIX A

AERODYNAMIC HEATING AND RADIATION EQUILIBRIUM CALCULATIONS

The radiation equilibrium temperature is that resulting from a balance between convective aerodynamic heat input, \dot{Q}_{AC} , and radiant heat loss, \dot{Q}_{R} . According to the Stefan-Boltzman law,

$$\dot{Q}_{R} = \varepsilon \sigma T_{W}^{4} \tag{1}$$

WHERE

E = EMMISSIVITY

STEFAN-BOLTZMAN CONSTANT

 = 0.480 x 10⁻¹²BTU (sec FT² ∘R⁴)

Tw = ABSOLUTE TEMPERATURE OF THE BODY SURFACE, R

THE AERODYNAMIC HEAT FLUX IS BASED ON STAGNATION POINT VALUES FOR A HEMISPHERICAL NOSE WHOSE RADIUS EQUALS THAT OF THE PANEL LENGTH. THE DATA IS BASED ON THEORY AND EXPERIMENTS OF REFERENCES 8 TO 10 WHICH ARE REVIEWED IN REFERENCE 7. IN REFERENCES 6 AND 8, AN APPROXIMATION IS GIVEN TO THE RESULTS OF REFERENCE 8, WHICH IS GIVEN BY THE FOLLOWING EQUATION. THIS EQUATION CONTAINS IMPLICITLY THE HYPERSONIC COLD WALL ASSUMPTION, BUT IS ADEQUATE FOR THE HIGH SPEEDS OF INTEREST (SEE REFERENCE 7).

$$\dot{Q}_{AC} = \frac{17,600}{\sqrt{R}} \left(\frac{V}{26,000} \right)^{3.15} \left(\frac{\rho}{.002378} \right)^{1/2}$$
 (2)

WHERE

GAC = AERODYNAMIC CONVECTIVE HEAT FLUX BTU/SEC FT2

R = LEADING EDGE RADIUS FOR AN EQUIVALENT HEMISPHERE, FT.

V = FLIGHT SPEED, FT/SEC

 ρ = DENSITY OF AMBIENT AIR, SLUGS/FT³

An equivalent leading edge radius of 18 inches has been used in connection with the heat shield heat flux calculations, which is reasonable for a typical heat shield in which the 18-inch panel might be an element. The 18-inch radius of curvature used here is the radius of curvature associated with the nose of the vehicle in which the heat shield panel is an element. This radius of curvature is not necessarily equal to the curvature of the individual panel; e.g., in the case of the flat panels. For example, the curved structural panels have a 54-inch radius and the curved heat shield

PANELS HAVE A RADIUS OF 30-INCHES FOR THOSE SPECIMENS TO BE MANUFACTURED AS CURVED PANELS. THESE LATTER CONSIDERATIONS DO NOT CONFLICT WITH THE POSSIBILITY OF HAVING AN EFFECTIVE 18-INCH RADIUS OF CURVATURE AT THE NOSE OF THE VEHICLE USING 18-INCH PANELS. EQUATIONS (1) AND (2), USING R = 1.5 ft. Show that the Highest Equilibrium temperature of the "SKIP-GLIDE-ONCE-AROUND" TRAJECTORY IS 2400°F. (ALTITUDE 260,000 ft., VELOCITY 20,000 ft/SEC, EMMISSIVITY, & = .8, FROM REFERENCE 1). THIS OF COURSE SHOWS THAT REFRACTORY METALS ARE ADEQUATE FOR WITHSTANDING THE PEAK TEMPERATURES OF THE RELATIVELY SLOWER HEATING RATES OF THE "SKIP-GLIDE-ONCE-AROUND EARTH" TRAJECTORY (ASSUMING TO THE PRESENCE OF AN ADEQUATE OXIDATION COATING.) THE DURATION OF THIS PEAK HEAT FLUX IN THE SKIP-GLIDE TRAJECTORY OF REFERENCE 1 IS OF THE ORDER OF TWENTY MINUTES. HOWEVER, IF THE VEHICLE POSSESSED A HEAT SHIELD WHOSE NOSE RADIUS WAS, SAY, 6 FEET COMPARED TO THE PRESENT 1.5 FEET, THEN THE MAXIMUM RADIATION EQUILIBRIUM TEMPERATURE WOULD BE OF THE ORDER OF 1900°F, AND THIS IS EASILY WITHIN THE CAPABILITY OF THE REFRACTORY METALS.

EQUATIONS (1) AND (2) ARE THE BASIS OF ALL SUBSEQUENT RADIATION AND AERODYNAMIC HEAT FLUX CALCULATIONS FOR THE ESCAPE VELOCITY RE-ENTRY TRAJECTORY.

APPENDIX B

TEMPERATURE RESPONSE OF HEAT SHIELD AND STRUCTURAL PANEL FOR A SPECIFIED EXTERNAL HEAT INPUT

APPENDIX B: Temperature Response of Heat Shield and Structural Panel for a Specified External Heat Input

The purpose of this study was to find the temperatures and temperature differentials developed in the heat shield and structural panel of a space vehicle for a given external heat flux history. The effect of thermal insulation between the shield and panel was investigated.

Theory

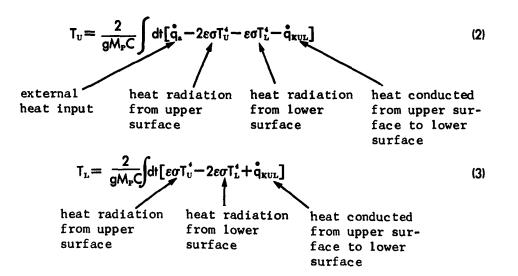
The heat shield and the structural panel were each approximated by homogeneous slabs of uniform temperature in order to find a mean temperature as a function of time. Then with the mean temperatures known the temperature differential across the slab was calculated. By using the differential temperatures, it was shown that the first approximation of the panel and shield as uniform temperature slabs was a reasonable one. The analysis is one dimensioned and neglects edge heat losses.

Case I: No Thermal Insulation Between the Heat Shield and the Structural Panel - Aerodynamic Heating

To obtain the mean heat shield temperature the following equation was used:

$$C\rho g \left[Z_c \frac{A_c}{A_w} + 2t_t \right] \frac{dT_{MHS}}{dt} = \dot{q}_a(t) - 2\epsilon\sigma T_{MHS}^4$$
"Thermal Mass" rate of rate of of heat shield external heat heat input radiated

To find the difference between the upper surface temperature T_L , and the lower surface temperature, T_L , of the heat shield, the upper and lower halves are analyzed as separate slabs below.



Temperature Difference $\Delta T = T_v - T_r = \theta$

$$\Delta T = T_{U} - T_{L} = T_{U}(0) - T_{L}(0) - \frac{2}{gM_{P}C} \int dt \left[\dot{q}_{a} - 3\varepsilon\sigma T_{U}^{4} + 3\varepsilon\sigma T_{L}^{4} - 2\dot{q}_{KUL} \right]$$
(4)

Approximations for $(\Delta T/2T_M)^2 \le .01$; $|\Delta T/T_M| \le .2$

$$\mathsf{T}_{\mathsf{U}}^{4} = \mathsf{T}_{\mathsf{M}}^{4} \left(1 + \frac{\Delta \mathsf{T}}{2\mathsf{T}_{\mathsf{M}}} \right)^{4} = \mathsf{T}_{\mathsf{M}}^{4} \left(1 + \frac{2\Delta \mathsf{T}}{\mathsf{T}_{\mathsf{M}}} \right) \tag{5}$$

$$\mathsf{T}_{L}^{4} = \mathsf{T}_{M}^{4} \left(1 - \frac{\Delta \mathsf{T}}{2\mathsf{T}_{M}} \right)^{4} = \mathsf{T}_{M}^{4} \left(1 - \frac{2\Delta \mathsf{T}}{\mathsf{T}_{M}} \right) \tag{6}$$

From equations (5) and (6)

$$-3\varepsilon\sigma T_{U}^{4} + 3\varepsilon\sigma T_{L}^{4} = 3\varepsilon\sigma T_{M}^{4} \left[-\left(1 + \frac{2\Delta T}{T_{M}}\right) + \left(1 - \frac{2\Delta T}{T_{M}}\right) \right]$$

$$= -12\varepsilon\sigma T_{M}^{3} \Delta T \tag{7}$$

From Fouriers' Law (Ref. 7, p. 7, and noting that Ac/Aw is the solid area fraction of the honeycomb core)

$$\dot{q}_{KUL} = k_{SP} \frac{Ac}{Aw} \frac{\Delta T}{\Delta Z_{SP}} \tag{8}$$

From (4), (7) and (8)

$$\frac{d\Delta T}{dt} = \frac{2}{gM_{P}C} \dot{q}_{a} - \frac{2}{gM_{P}C} \left[\frac{2k_{SP}}{\Delta Z_{SP}} \frac{Ac}{Aw} + 12\epsilon\sigma T_{M}^{3} \right] \Delta T$$
can be calculated
for a given
trajectory
$$(9)$$

Equation 9 was solved for ΔT (or θ) as a function of time.

Case II: Aerodynamic Heating with Thermal Insulation Between the Heat Shield and the Structural Panel Basis of data presented

To find the mean temperature of the structural panel, the heat shield and the insulation, three equations were used simultaneously.

Heat Shield:

$$(gM_{P}C)_{HS} \frac{dT_{MHS}}{dt} = \dot{q}_{a}(t) - 2\varepsilon\sigma T_{MHS}^{4} + \varepsilon\sigma T_{I}^{4} - \frac{2k_{I}}{\Delta Z_{I}}(T_{MHS} - T_{I})$$
(10)

Structural Panel:

$$(gM_{P}C)_{SP} \frac{dT_{MSP}}{dt} = \frac{2k_{I}}{\Delta Z_{I}} (T_{I} - T_{MSP}) - 2\varepsilon\sigma T_{MSP}^{4} + \varepsilon\sigma T_{I}^{4}$$
(11)

Insulation:

$$(gMC)_{i} \frac{dT_{i}}{dt} = \frac{2k_{t}}{\Delta Z_{i}} (T_{MHS} + T_{MSP} - 2T_{i}) - 2\varepsilon\sigma T_{i}^{4} + \varepsilon\sigma T_{MSP}^{4} + \varepsilon\sigma T_{MHS}^{4}$$

$$+ \varepsilon\sigma T_{MHS}^{4}$$
(12)

Temperature Difference Across the Structural Panel

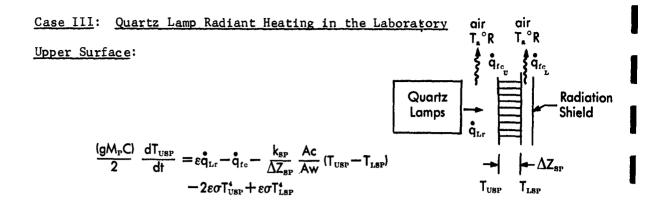
$$\frac{(gM_{P}C)}{2} \frac{dT_{USP}}{dt} = \frac{2k_{I}}{\Delta Z_{I}} (T_{I} - T_{MSP}) - 2\varepsilon\sigma T_{USP}^{4} + \varepsilon\sigma T_{LSP}^{4} - \frac{k_{SP}}{\Delta Z_{SP}} \frac{Ac}{Aw} (T_{USP} - T_{LSP}) + \varepsilon\sigma T_{I}^{4}$$
(13)

$$\frac{gM_{P}C}{2}\frac{dT_{LSP}}{dt} = \frac{k_{SP}}{\Delta Z_{SP}}\frac{Ac}{Aw}(T_{USP} - T_{LSP}) + \varepsilon\sigma T_{USP}^{4} - 2\varepsilon\sigma T_{LSP}^{4}$$
(14)

Equation (15) - Equation (13) - Equation (14), defines $\theta_{sp} = \Delta T_{sp} = T_{usp} - T_{Lsp}$

$$\frac{1}{2} (gM_{P}C) \frac{d\theta_{sP}}{dt} = \frac{2k_{I}}{\Delta Z_{I}} (T_{I} - T_{MSP}) - 3\varepsilon\sigma T_{USP}^{4} + 2\varepsilon\sigma T_{LSP}^{4} + \varepsilon\sigma T_{I}^{4}$$
$$-\frac{2k_{SP}}{\Delta Z_{SP}} (\theta_{SP})$$
(15)

The above equations were solved step-by-step finite difference method to yield the results depicted in Figures 3 to 9 in the text.



Lower Surface:

$$\frac{(gM_{P}C)}{2}\frac{dT_{LSP}}{dt} = \varepsilon\sigma T_{USP}^{4} - \varepsilon\sigma T_{LSP}^{4} + \frac{k_{SP}}{\Delta Z_{SP}}\frac{Ac}{Aw}(T_{USP} - T_{LSP})$$

where $q_{\rm fc}$ is the free convection heat loss/sq ft. to the air. For a vertical plate we have (Ref. 7, p. 241)

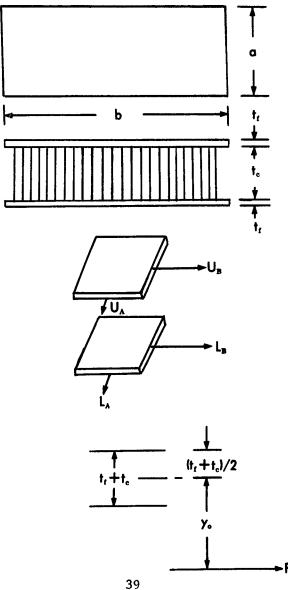
$$\dot{\mathbf{q}}_{fc_{_{_{\mathbf{U}}}}}(\mathbf{T}_{_{\mathbf{U}}}) = 0.2 (\mathbf{T}_{_{\mathbf{U}}} - \mathbf{T}_{_{\mathbf{a}}})^{1.25}$$

APPENDIX C

STRENGTH ANALYSIS OF HONEYCOMB PANELS
WITH THERMAL ECCENTRICITIES

APPENDIX C

Consider a structural panel of dimensions "a" and "b" with a lateral load of P pounds per lineal inch applied along the "A" side. This load is applied by a piston arrangement such that the load is maintained as a constant with no increase due to thermal expansion. It is assumed that the honeycomb panel can be analyzed as two mutually perpendicular beam strips which deform thermally and elastically in a sinusoidal shape while maintaining deflection compatibility at the center of the strips. It is further assumed that there exists a temperature gradient between the face sheets and a gradient between the center of the panel and the edge. Also, the temperature of the upper and lower face sheets are the same at the panel edge.



Summation Moments about U_B :

$$L_{B}(t_{t}+t_{c}) = P\left(y_{o} + \frac{t_{t}+t_{c}}{2}\right)$$

$$L_B = \sigma_{BL} t_t$$

$$P = \frac{\sigma_{BL} t_{t} (t_{t} + t_{c})}{y_{o} + \underline{t_{t} + t_{c}}}$$

Summation Moments About L_B:

$$U_{B}(t_{t}+t_{c})=-P\left(y_{o}-\frac{t_{t}+t_{o}}{2}\right)$$

$$U_{\rm\scriptscriptstyle B} = \sigma_{\rm\scriptscriptstyle BU} t_{\rm\scriptscriptstyle B}$$

$$P = \frac{-\frac{\sigma_{BU} t_t (t_t + t_c)}{y_c - \frac{t_t + t_c}{2}}}{\frac{\tau_c + \tau_c}{2}}$$

Summation Forces in "a" direction:

$$U_{\Lambda} = -L_{\Lambda}$$

$$\sigma_{\text{AU}} = -\sigma_{\text{AL}}$$

Thermal Expansion:

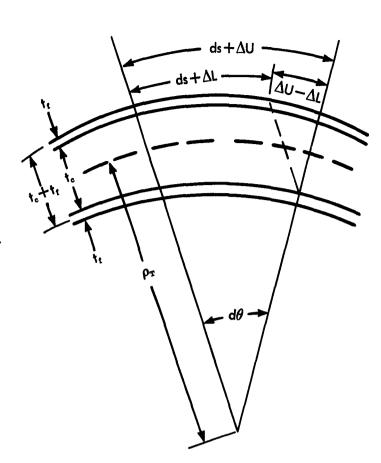
$$\Delta L = \alpha_L \Delta T_L ds$$

 $\Delta U = \alpha_U \Delta T_U ds$

$$d\theta = \frac{\Delta U - \Delta L}{t_t + t_c} = ds \frac{\alpha_U \Delta T_U - \alpha_L \Delta T_L}{t_t + t_c}$$

$$d\theta = \frac{ds + \frac{\Delta U + \Delta L}{2}}{\rho_{T}} \cong \frac{ds}{\rho_{T}}$$

$$\frac{1}{\rho_{\rm r}} = \frac{\alpha_{\rm U} \Delta T_{\rm U} - \alpha_{\rm L} \Delta T_{\rm L}}{t_{\rm t} + t_{\rm c}}$$



Pr = curvature due to differential thermal expansion

$$\rho_{\scriptscriptstyle \rm Ta}\!=\!\rho_{\scriptscriptstyle \rm Tb}\!=\!\rho_{\scriptscriptstyle \rm T}$$

Elastic Deformation:

$$\varepsilon_{U} = \frac{\sigma_{BU} - \mu \sigma_{AU}}{E_{U}} ds$$

$$\varepsilon_{L} = \frac{\sigma_{BL} - \mu \sigma_{AL}}{E_{L}} ds$$

$$d\theta = \frac{\varepsilon_{U} - \varepsilon_{L}}{t_{t} + t_{c}}$$

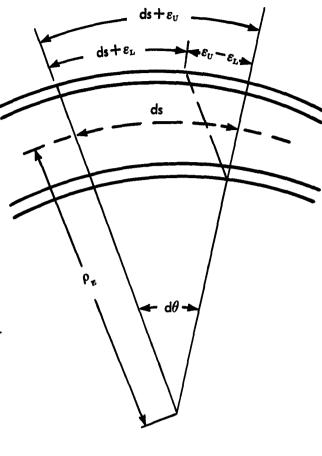
$$d\theta = \left(\frac{\sigma_{BU} - \mu \sigma_{AU}}{E_{U}} - \frac{\sigma_{BL} - \mu \sigma_{AL}}{E_{L}}\right) \frac{ds}{(t_{t} + t_{c})}$$

$$d\theta = \frac{ds}{\rho_{BE}}$$

$$\frac{1}{\rho_{AE}} = \frac{\sigma_{BU} - \mu \sigma_{AU}}{E_{U}} - \frac{\sigma_{BL} - \mu \sigma_{AL}}{E_{L}}$$

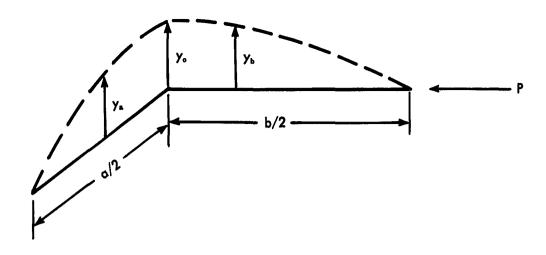
$$\frac{1}{t_{t} + t_{c}} = \frac{\sigma_{AU} - \mu \sigma_{BU}}{E_{U}} - \frac{\sigma_{AL} - \mu \sigma_{BL}}{E_{L}}$$

$$\frac{1}{t_{t} + t_{c}} = \frac{\sigma_{AU} - \mu \sigma_{BU}}{E_{U}} - \frac{\sigma_{AL} - \mu \sigma_{BL}}{E_{L}}$$



 ρ_{BE} = curvature due to elastic deformation in "b" direction

 ρ_{AE} = curvature due-to elastic deformation in "a" direction



Displacement of Two Beam Strips

General Form of Sine Curve (Assumed Shape)

$$y=y_0 \sin \frac{\pi X}{P}$$

$$y^{II} = \frac{\pi^2}{2} y_o \sin \frac{\pi \chi}{2}$$

and

$$y'' = \frac{M}{EI} = \frac{1}{\rho}$$

Combined Curvature:

$$\frac{1}{\rho_{\text{B}}} = \frac{1}{\rho_{\text{BT}}} + \frac{1}{\rho_{\text{BE}}} = \frac{\alpha_{\text{U}} \Delta T_{\text{U}} - \alpha_{\text{L}} \Delta T_{\text{L}} + \frac{\sigma_{\text{BU}} - \mu \sigma_{\text{AU}}}{E_{\text{U}}} - \frac{\sigma_{\text{BL}} - \mu \sigma_{\text{AL}}}{E_{\text{L}}}}{t_{\text{f}} + t_{\text{c}}}$$

$$\frac{1}{\rho_{A}} = \frac{1}{\rho_{AT}} + \frac{1}{\rho_{AE}} = \frac{\alpha_{U}\Delta T_{U} - \alpha_{L}\Delta T_{L} + \frac{\sigma_{AU} - \mu\sigma_{BU}}{E_{U}} - \frac{\sigma_{AL} - \mu\sigma_{BL}}{E_{L}}}{t_{L} + t_{c}}$$

Substituting $1/\rho$ into "y" equation for a sine wave at $X = \sigma/2$ and b/2respectively.

$$\frac{1}{\rho_{A}} = \frac{\pi^{2}}{\alpha^{2}} y_{oA} \qquad \qquad \frac{1}{\rho_{B}} = \frac{\pi^{2}}{b^{2}} y_{oB}$$

$$\frac{1}{\rho_{\rm B}} = \frac{\pi^2}{b^2} y_{\rm ol}$$

$$y_{oA} = \frac{a^2}{\pi^2 \rho_A}$$

$$y_{oB} = \frac{b^2}{\pi^2 \rho_B}$$

For continuity:

$$y_{oA} = y_{oB}$$

$$\frac{a^2}{\pi^2 \rho_A} = \frac{b^2}{\pi^2 \rho_B}$$

Substituting for ρ_A and ρ_B

$$(b^2 - a^2) (\alpha_U \Delta T_U - \alpha_L \Delta T_L) + (b^2 + \mu a^2) \left(\frac{\sigma_{BU}}{E_U} - \frac{\sigma_{BL}}{E_L} \right) = (a^2 + \mu b^2) \left(\frac{\sigma_{AU}}{E_U} - \frac{\sigma_{AL}}{E_L} \right)$$

From equilibrium conditions:

$$\sigma_{\scriptscriptstyle \mathrm{AU}} = -\sigma_{\scriptscriptstyle \mathrm{AL}}$$

$$\sigma_{AU} = -\sigma_{AL} = \frac{(b^2 - a^2) (\alpha_U \Delta T_U - \alpha_L \Delta T_L) + (b^2 + \mu a^2) \left(\frac{\sigma_{BU}}{E_U} - \frac{\sigma_{BL}}{E_L}\right)}{(a^2 + \mu b^2) \left(\frac{1}{E_U} + \frac{1}{E_L}\right)}$$

Substituting σ_{AU} into y_{oB} to obtain y_o at panel center

$$y_o = \frac{\alpha^2 b^2 (1 + \mu)}{\pi^2 (t_t + t_c) (\alpha^2 + \mu b^2)} \left\{ (\alpha_U \Delta T_U - \alpha_L \Delta T_L) + (1 - \mu) \left(\frac{\sigma_{BU}}{E_U} - \frac{\sigma_{BL}}{E_L} \right) \right\}$$

From equilibrium considerations:

$$\sigma_{\rm BL} t_{\rm f} + \sigma_{\rm BU} t_{\rm f} = P$$

For lower skin failure at $\sigma_{\scriptscriptstyle L}$

$$\sigma_{BU} = \frac{P}{t_f} - \sigma_L$$

$$\gamma_o = \frac{\alpha^2 b^2 (1 + \mu)}{\pi^2 (t_f + t_c) (\alpha^2 + \mu b^2)} \left\{ \alpha_U \Delta T_U - \alpha_L \Delta T_L + (1 - \mu) \left[\frac{P}{t_f E_U} - \sigma_L \left(\frac{1}{E_U} + \frac{1}{E_L} \right) \right] \right\}$$

For lower skin failure

$$P = \frac{\sigma_{\rm L} t_{\rm f} (t_{\rm f} + t_{\rm c})}{\gamma_{\rm o} + \frac{t_{\rm f} + t_{\rm c}}{2}}$$

Substituting Yo into above:

$$\begin{cases} \frac{\alpha^{2}b^{2}(1-\mu^{2})}{\pi^{2}E_{U}t_{t}(t_{t}+t_{c})(\alpha^{2}+\mu b^{2})} \end{cases} P^{2} + \\ \begin{cases} \frac{\alpha^{2}b^{2}(1+\mu)}{\pi^{2}(t_{t}+t_{c})(\alpha^{2}+\mu b^{2})} \left[\alpha_{U}\Delta T_{U} - \alpha_{L}\Delta T_{L} - (1-\mu)\sigma_{L}\left(\frac{1}{E_{U}} + \frac{1}{E_{L}}\right) \right] + \frac{t_{t}+t_{c}}{2} \end{cases} P \\ - \left\{ \sigma_{L}t_{t}(t_{t}+t_{c}) \right\} = O \end{cases}$$

Solution of the above equation yields a minimum negative P to fail the lower sheet. This P is one of the two solutions of the above equation and is only applicable if the lower sheet is the failure element. It is therefore necessary to also check for upper sheet failure.

For upper skin failure at σ_v

$$\sigma_{BL} = \frac{P}{t_t} - \sigma_{U}$$

$$\gamma_o = \frac{\alpha^2 b^2 (1 + \mu)}{\pi^2 (t_t + t_o) (\alpha^2 + \mu b^2)} \left\{ \alpha_{U} \Delta T_{U} - \alpha_{L} \Delta T_{L} + (1 - \mu) \left[\sigma_{U} \left(\frac{1}{E_{U}} + \frac{1}{E_{L}} \right) - \frac{P}{t_t E_{L}} \right] \right\}$$

For upper skin failure

$$P = \frac{-\sigma_{\rm u}t_{\rm f}(t_{\rm f}+t_{\rm c})}{y_{\rm o}-\frac{t_{\rm f}+t_{\rm c}}{2}}$$

Substituting yo into above:

$$\left\{ -\frac{\alpha^{2}b^{2}(1-\mu^{2})}{\pi^{2}(t_{t}+t_{c})(\alpha^{2}+\mu b^{2})t_{t}E_{L}} \right\} P^{2} + \\ \left\{ \frac{\alpha^{2}b^{2}(1+\mu)}{\pi^{2}(t_{t}+t_{c})(\alpha^{2}+\mu b^{2})} \left[\alpha_{U}\Delta T_{U} - \alpha_{L}\Delta T_{L} + (1-\mu)\sigma_{U} \left(\frac{1}{E_{U}} + \frac{1}{E_{L}} \right) \right] - \frac{t_{t}+t_{c}}{2} \right\} P \\ + \left\{ \sigma_{U}t_{t}(t_{t}+t_{c}) \right\} = O$$

Solution of the above equation yields the minimum negative P to fail the upper sheet. The minimum failure loads for both sheets are compared and the lowest of these two becomes the failure load for the panel.

To eliminate the need for solving a quadratic in P it is possible to make the following substitution:

$$\sigma_{\rm BU} = \frac{P}{A} - \frac{Mc}{I}$$

$$\sigma_{\rm BL} = \frac{P}{A} + \frac{Mc}{I}$$

$$\sigma_{\rm BL} = \sigma_{\rm U} + \frac{2Mc}{l}$$

For upper sheet failure

$$\sigma_{\rm BU} = \sigma_{\rm L} - \frac{2Mc}{I}$$

For lower sheet failure

$$\frac{1}{\rho_{\rm B}} = \frac{M}{E^{\rm i}l}$$

$$\frac{E^{1}c}{\rho_{B}} = \frac{Mc}{I}$$

$$\frac{1}{\rho_{\rm B}} = \frac{\pi^2}{b^2} \, \gamma_{\rm o}$$

$$\frac{E^1c}{\rho_R} = \frac{E^1c\pi^2\gamma_o}{b^2}$$

$$E^{1} = \frac{2E_{U}E_{L}}{E_{U} + E_{L}}$$

$$c = \frac{t_t + t_c}{2}$$

$$\frac{E^{1}c}{\rho_{B}} = \frac{E_{U}E_{L}\pi^{2}\gamma_{o}(t_{f}+t_{c})}{(E_{U}+E_{L})b^{2}}$$

For lower sheet failure

$$\left(\frac{\sigma_{\text{BU}}}{E_{\text{U}}} - \frac{\sigma_{\text{BL}}}{E_{\text{L}}}\right) = \frac{\sigma_{\text{L}}}{E_{\text{U}}} - \frac{2M_{\text{C}}}{1E_{\text{U}}} - \frac{\sigma_{\text{L}}}{E_{\text{L}}}$$

$$\left(\frac{\sigma_{\text{BU}}}{E_{\text{U}}} - \frac{\sigma_{\text{BL}}}{E_{\text{L}}}\right) = \sigma_{\text{L}} \left(\frac{1}{E_{\text{U}}} - \frac{1}{E_{\text{L}}}\right) - \frac{2E_{\text{L}}\pi^{2}y_{\text{o}}(t_{t} + t_{\text{c}})}{(E_{\text{U}} + E_{\text{I}})b^{2}}$$

For upper sheet failure

$$\left(\frac{\sigma_{\text{BU}}}{E_{\text{U}}} - \frac{\sigma_{\text{BL}}}{E_{\text{L}}}\right) = \sigma_{\text{U}}\left(\frac{1}{E_{\text{U}}} - \frac{1}{E_{\text{L}}}\right) - \frac{2E_{\text{U}}\pi^{2}y_{\circ}(t_{t} + t_{c})}{(E_{\text{U}} + E_{\text{L}})b^{2}}$$

For lower sheet failure

$$y_{o} = \frac{\alpha^{2}b^{2}(1 + \mu) (E_{U} + E_{L})}{\pi^{2}(t_{L} + t_{o})[(\alpha^{2} + \mu b^{2}) (E_{U} + E_{L}) + \alpha^{2}(1 - \mu^{2})2E_{L}]} \left\{ \alpha_{U}\Delta T_{U} - \alpha_{L}\Delta T_{L} + (1 - \mu)\sigma_{L}\left(\frac{1}{E_{U}} - \frac{1}{E_{L}}\right) \right\}$$

For upper sheet failure

$$y_{o} = \frac{\alpha^{2}b^{2}(1+\mu)(E_{U}+E_{L})}{\pi^{2}(t_{t}+t_{c})[(\alpha^{2}+\mu b^{2})(E_{U}+E_{L})+\alpha^{2}(1-\mu^{2})2E_{U}]} \left\{ \alpha_{U}\Delta T_{U} - \alpha_{L}\Delta T_{L} + (1-\mu)\sigma_{U}\left(\frac{1}{E_{U}} - \frac{1}{E_{L}}\right) \right\}$$

Substituting the y from above based on the proper face sheet failure into the P equations of Page 37 and accepting the minimum negative P will give the critical compressive load to fail the panel.

Due to the assumption of an effective modulus for the section, these equations are modified as follows:

ΣM about U_m

$$P = \frac{\sigma_{BL}t_{t}(t_{t}+t_{c})}{y_{o} + \left(\frac{E_{L}}{E_{U}+E_{L}}\right)(t_{t}+t_{c})}$$

ΣM about L

$$P = \frac{-\sigma_{BU}t_{t}(t_{t}+t_{c})}{y_{o} - \left(\frac{E_{U}}{E_{U}+E_{L}}\right)(t_{t}+t_{c})}$$

APPENDIX D

1

ILLUSTRATIONS

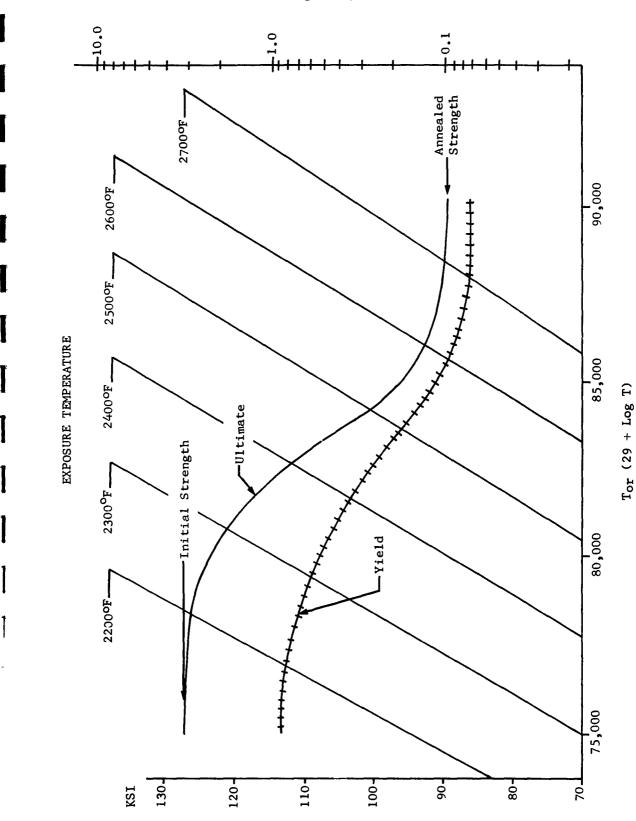


FIGURE I ULTIMATE AND YIELD STRENGTH AT ROOM TEMPERATURE VS THERMAL EXPOSURE PARAMETER FOR COLD-ROLLED TZM MOLYBDENUM SHEET

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TIME TO TEMP: 20 SECONDS SOAK TIME: 90 SECONDS

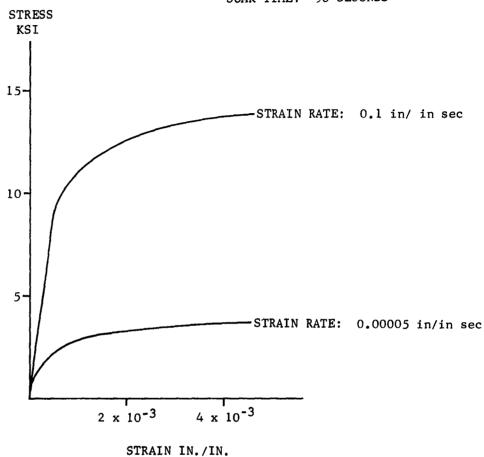


FIGURE 2 EFFECT OF STRAIN RATE ON THE STRESS-STRAIN CHARACTERISTICS OF ARC-CAST MOLYBDENUM SHEET WHEN TESTED AT 3000°F IN ARGON ATMOSPHERE

(Reference: DMIC Report No. 140)



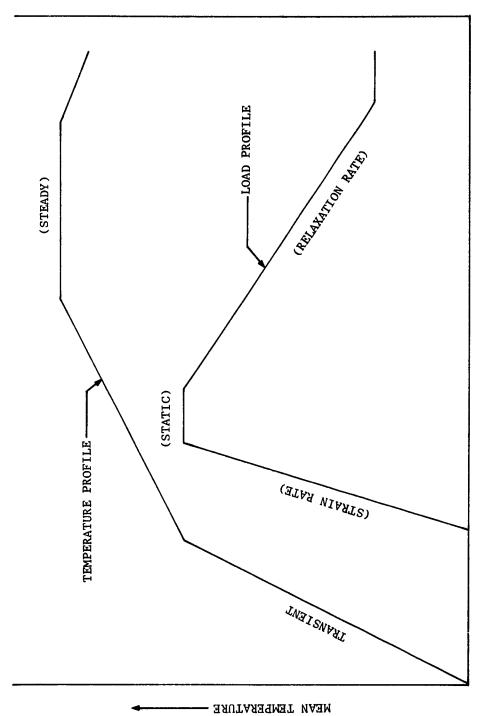


FIGURE 3 MEAN TEMPERATURE AND LOAD RATE PROFILE OF HEAT SHIELDS VS TIME FOR A RE-ENTRY TRAJECTORY

TIME -

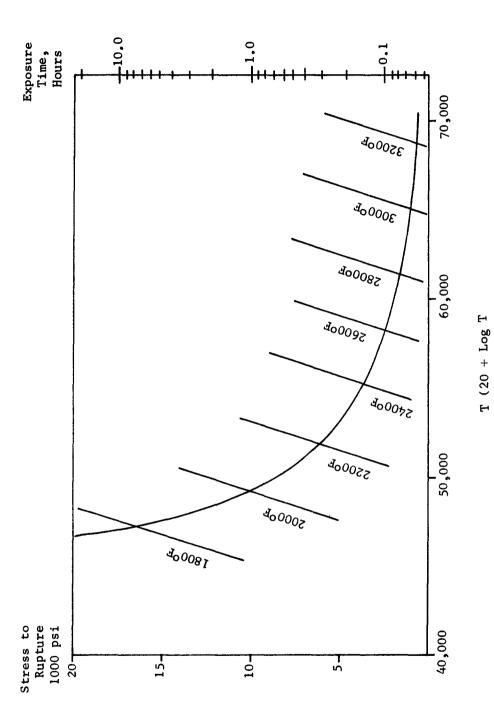
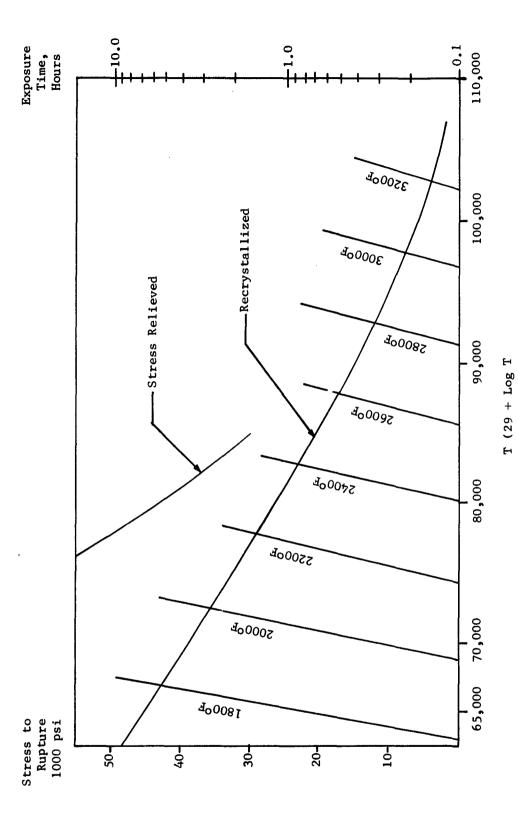
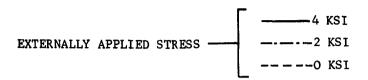


FIGURE 4 STRESS-TO-RUPTURE STRENGTH AT VARIOUS TEMPERATURES VS THERMAL EXPOSURE PARAMETER, D-36 COLUMBIUM



72.

FIGURE 5 STRESS-TO-RUPTURE STRENGTH AT VARIOUS TEMPERATURES VS THERMAL EXPOSURE PARAMETER, TZM MOLYBDENUM



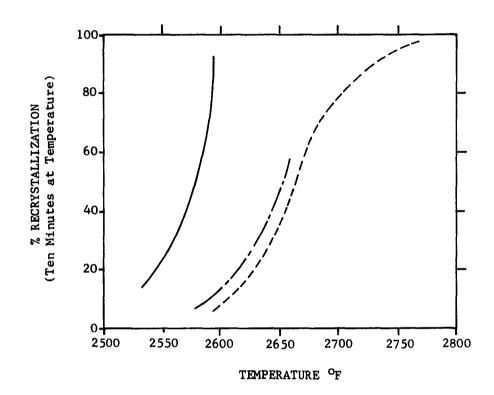
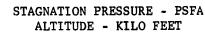


FIGURE 6 EFFECT OF APPLIED STRESS ON RATE OF RECRYSTALLIZATION VS TEMPERATURE ON 90.1% REDUCED TZM MOLYBDENUM



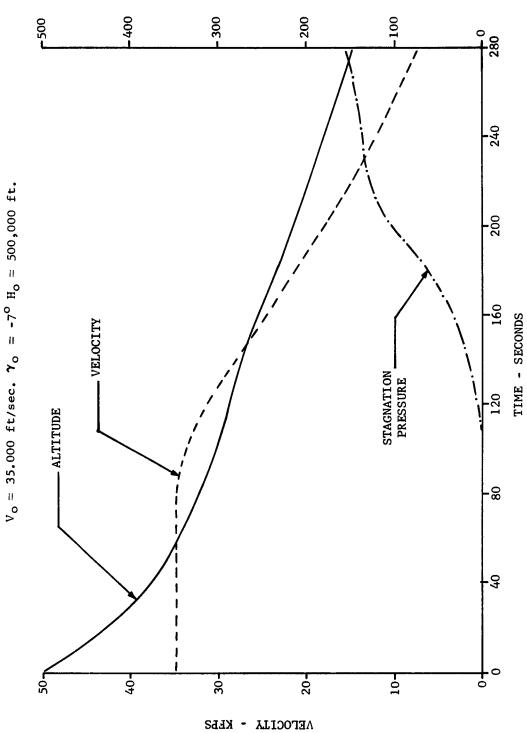


FIGURE 7 VELOCITY, ALTITUDE, AND STAGNATION PRESSURE VS TIME FOR AN ESCAPE VELOCITY RE-ENTRY TRAJECTORY

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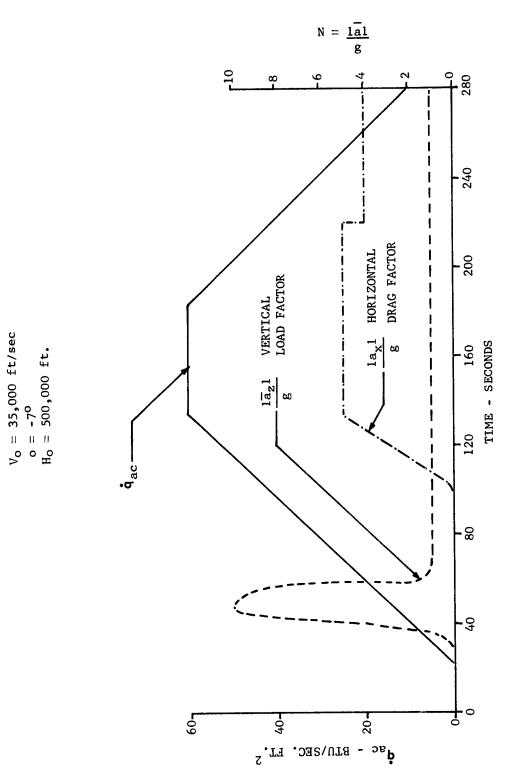
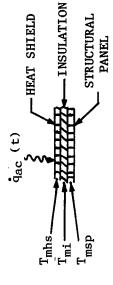


FIGURE 8 AERODYNAMIC HEAT INPUT (q), AND ACCELERATION LOAD FACTOR VS TIME FOR ESCAPE VELOCITY RE-ENTRY



 $\frac{2 \text{ Kins}}{\Delta \text{ Zins}} = 1.28 \times 10^{-2}$ 9° = ∃

ESCAPE VELOCITY REENTRY TRAJECTORY

$$V_o = 35,000 \text{ ft/sec. } \gamma_o = -7^0 \text{ H}_o = 500,000 \text{ ft.}$$

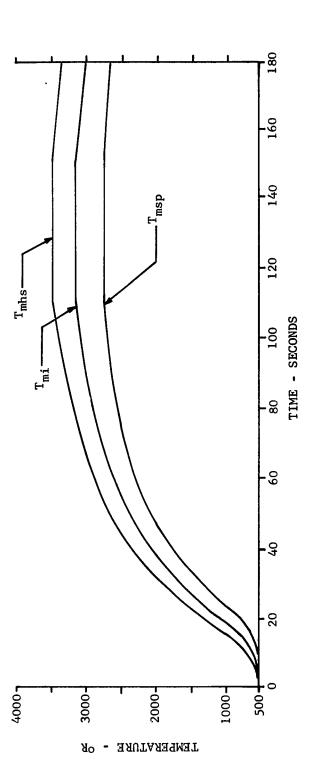
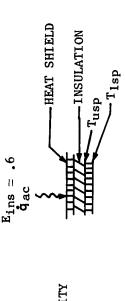


FIGURE 9 MEAN TEMPERATURE RESPONSE FOR IDEALIZED HEAT SHIELD, INSULATION AND STRUCTURAL PANEL

-HEAT SHIELD -INSULATION $E_{ins} = .6$ - .0163, $\frac{2~Kins}{\Delta~Zins} = 1.28~\times~10^{-2}~\frac{Btu}{sec.ft^2} o$ Based on mean temperature histories of figure 3 for escape velocity referry trajectory $\Delta Z_{sp} = .04 \text{ ft.}, \frac{Ac}{Aw}$ $V_{o} = 35,000 \text{ ft/sec.}, \text{ Yo} = 7^{\circ}, \text{ H}_{o} = 500,000 \text{ ft.}$ Cb D-36, $K_{Sp} = 1.167 \times 10^{-2} \frac{B_{tu}}{\text{sec.ft}^{20} F/Ft}$





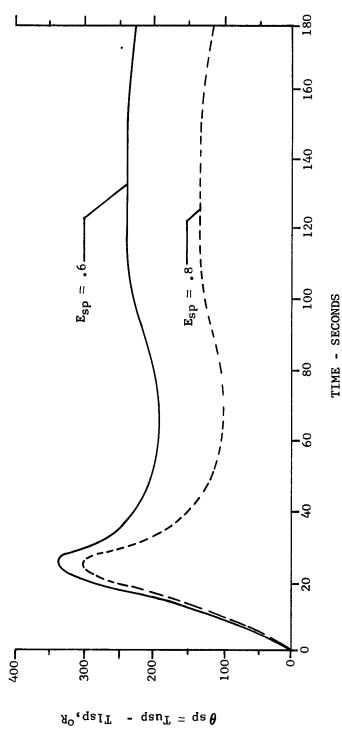
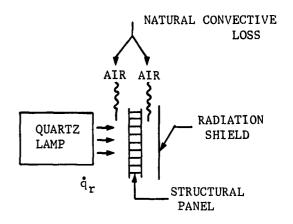


FIGURE 10 STRUCTURAL PANEL PLATE DIFFERENTIAL TEMPERATURE, $oldsymbol{ heta}$, VS TIME

- $qsu^T = qs\theta$



E = .8

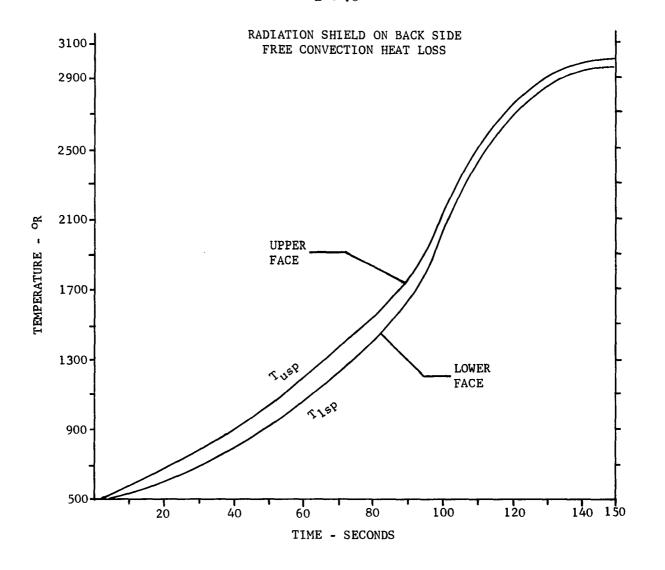


FIGURE 11 STRUCTURAL PANEL TEMPERATURE VS TIME - LAB TEST (SLOW HEATING SCHEDULE)

RADIATION SHIELD FOR BACK SIDE OF PANEL FREE CONVECTIVE LOSSES FROM BOTH SIDES SLOW HEATING SCHEDULE

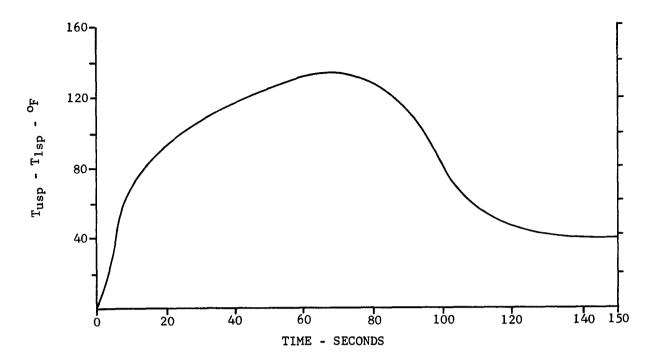


FIGURE 12a TEMPERATURE DIFFERENTIAL FOR STRUCTURAL PANEL - LABORATORY TEST

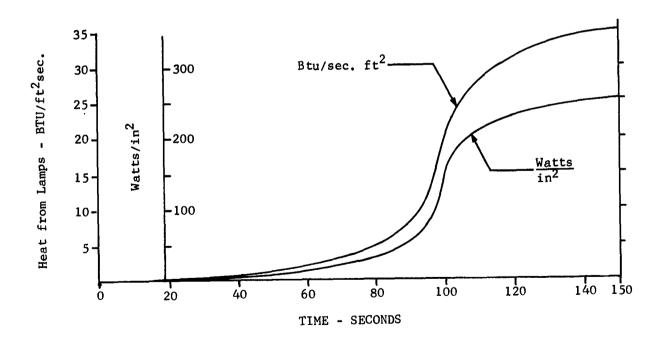
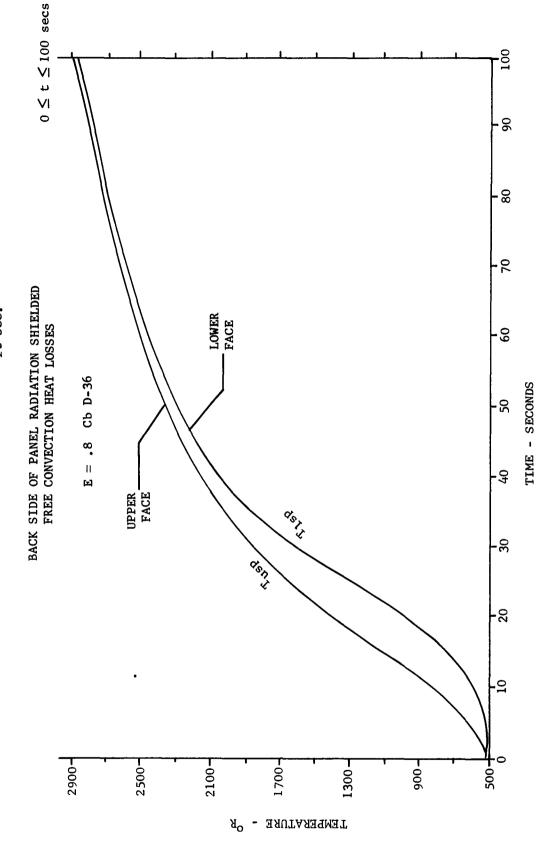


FIGURE 12b REQUIRED RATE OF HEAT OUTPUT FROM LAMPS VS TIME (SLOW HEATING SCHEDULE)

FIGURE 13 STRUCTURAL PANEL TEMPERATURE VS TIME - LABORATORY TEST (RAPTD HEATING SCHEDULE)







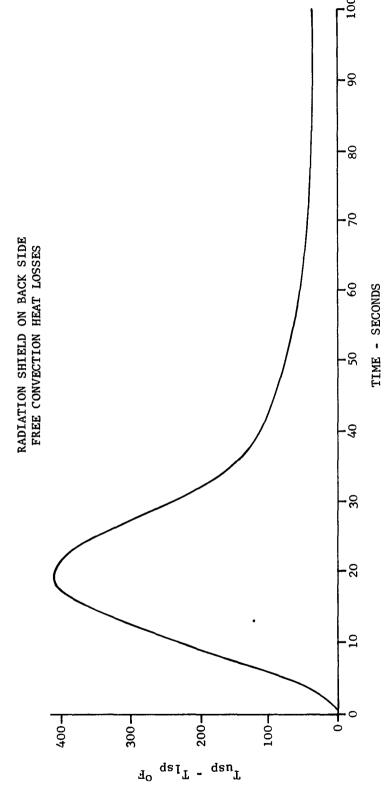


FIGURE 14 STRUCTURAL PANEL DIFFERENTIAL TEMPERATURE - (RAPID HEATING SCHEDULE)

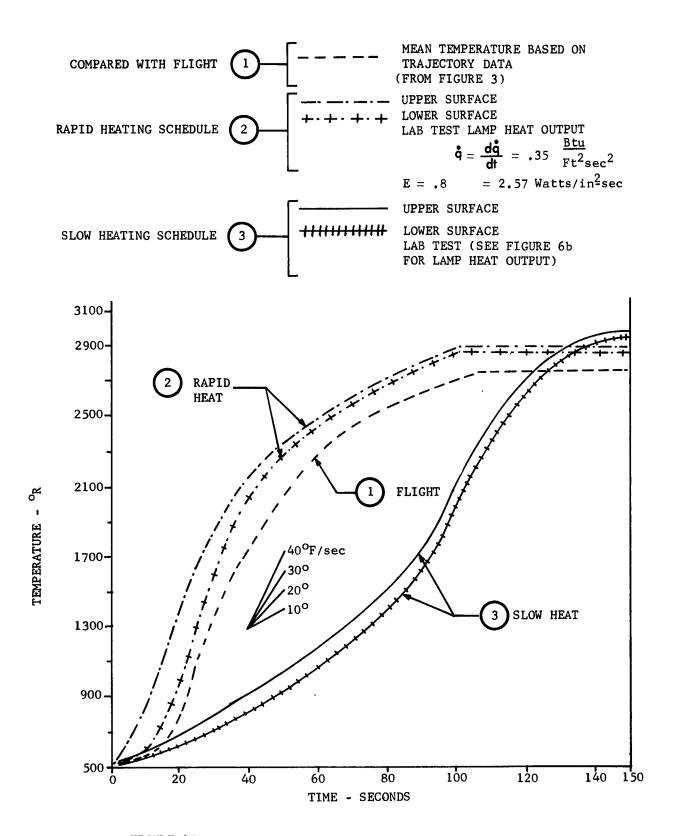


FIGURE 15 STRUCTURAL PANEL TEMPERATURE VS TIME FOR LABORATORY HEATING SCHEDULES AND COMPARED WITH FLIGHT

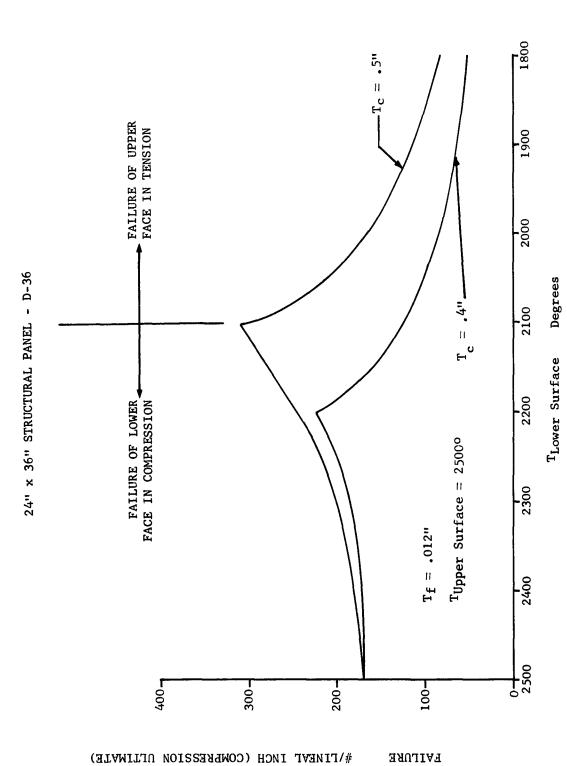


FIGURE 16 ULTIMATE COMPRESSIVE FAILURE VS TEMPERATURE DIFFERENTIAL OF D-36 COLUMBIUM

-009

#\rINEVL INCH (COMPRESSION ULTIMATE)

₽008

FAILURE OF UPPER FACE IN TENSION

24" x 36" STRUCTURAL PANEL - TZM

FIGURE 17 ULTIMATE COMPRESSIVE FAILURE VS TEMPERATURE DIFFERENTIAL OF TZM MOLYBDENUM

2000

2100

2300

2500

 $^{TUpper\ Surface} = 2500^o$

200-

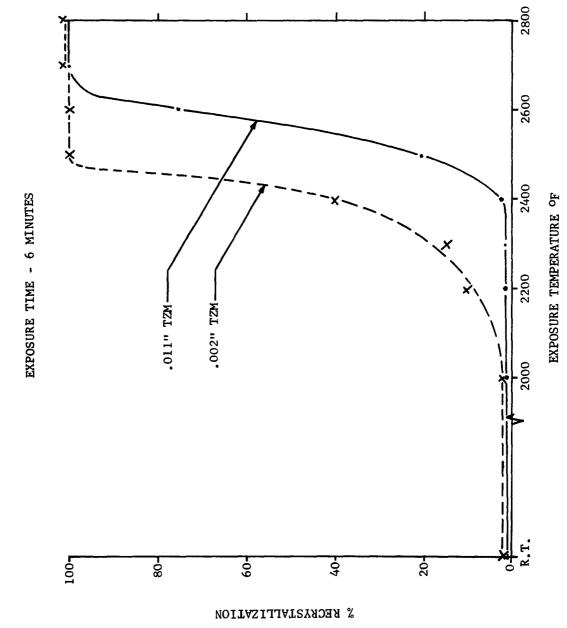
FAILURE

Degrees

2200 T_Lower Surface

400-

FIGURE 18 RECRYSTALLIZATION BEHAVIOR OF .011 AND .002 TZM (GRAPH)



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2500°F, 6 min.



2400°F, 6 min.

As-Received



2700°F, 6 min.





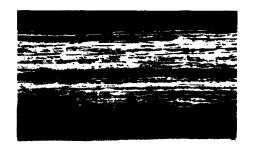
2600°F, 6 min.



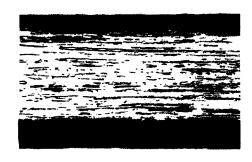
10 gm. K₃Fe(CN)₆ 10 gm. NaOH 200 ml. H₂O Etchant:



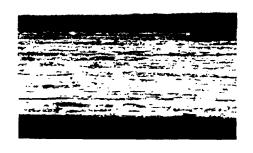
FIGURE 19: MICROSTRUCTURES OF .011" TZM RECRYSTALLIZATION SPECIMENS



As Received



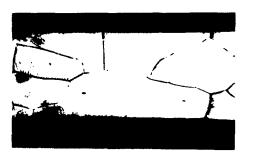
 2200° F, 6 min.



2300°F, 6 min.



 $2400^{\circ}F$, 6 min.

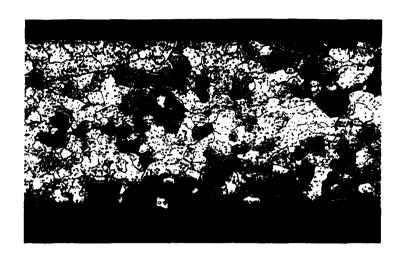


Mag. 500X

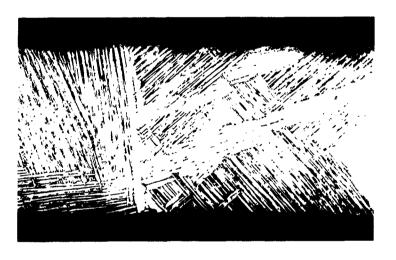
2600°F, 6 min.

Etchant: 10 gm K₃Fe(CN)₆ 10 gm. NaOH 200 ml H₂O

FIGURE 20: MICROSTRUCTURES OF .002" TZM RECRYSTALLIZATION SPECIMENS



As-Received



Held 2 minutes at 2900°F in argon

Mag. 100X

Etchant: 1 HF 2 HNO₃ 97 H₂O

FIGURE 21: MICROSTRUCTURES OF .012" TITANIUM SHEET



Specimen T11 exposed 10 minutes at $2400^{\circ}F$ in air



Specimen T12 exposed 10 minutes at 2600°F in air

Mag. 100X

Etchant: 25 HF 12.5 HNO3 12.5 H₂SO₄ 50 H₂O

FIGURE 22: MICROSTRUCTURES OF .012" TITANIUM SHEET COSTED WITH .004" $\rm A1_2O_3-2.5TiO_2$ AND .002" A1

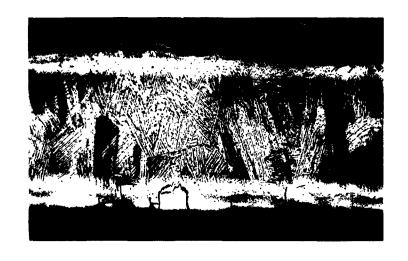
 $\begin{array}{c} \text{Specimen TG} \\ \text{exposed 2 minutes at 2400} \\ \text{OF in argon} \end{array}$

Mag 100X

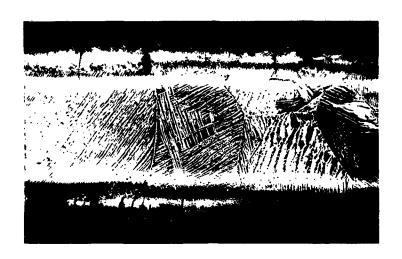
Etchant: 25HF

12.5 HNO₃ 12.5 H₂SO₄ 50 H₂O

FIGURE 23: MICROSTRUCTURE OF .012" TITANIUM SHEET COATED WITH .004" A1 0 3-2.5TiO 2



Specimen T28 exposed 10 minutes at 2400°F in air



Specimen T26 exposed 10 minutes at $2600^{\rm O}$ in air

Mag. 75X

Etchant: 25 HF 12.5 HNO₃ 12.5 H₂SO₄ 50 H₂O

FIGURE 24: MICROSTRUCTURES OF .032" TITANIUM SHEET COATED WITH .003" Cr AND .003" A1

X600

FIGURE 25 2 MIL D-36 BONDED WITH 60 (10⁻⁶)
IN. TITANIUM AT 1500°F -10 MIN.



600X

200X

FIGURE 26a 2 MIL TZM BONDED WITH TITANIUM HYDRIDE AT 1600°F UNETCHED

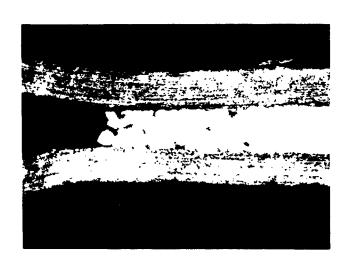
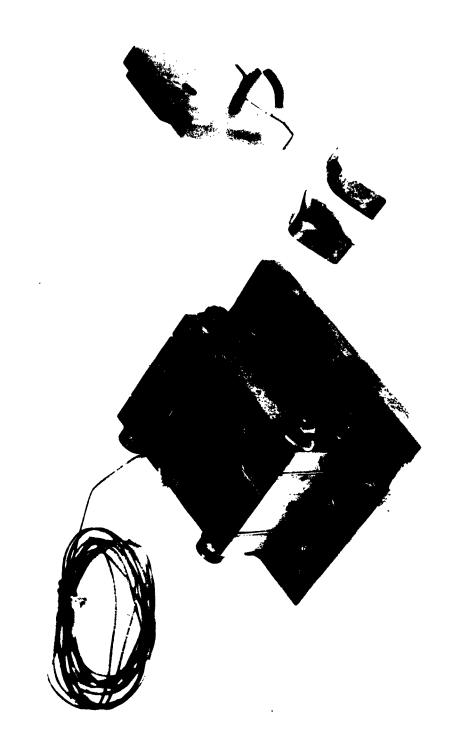


FIGURE 26b 2 MIL TZM BONDED WITH TITANIUM HYDRIDE AT 1600° FOR 10 MIN. MURAKAMIS ETCH





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APPENDIX E

Marine Same

TABLES

TABLE 1

T

EVALUATION OF PRE-BRAZE CLEANING PROCEDURES FOR TZM MOLYBDENUM ALLOYA

CLEANING Procedure	COMPOSITION (VOL. %-BAL. H20)	IMMER TEMP.	ળજી	ON TIME	INITIAL WTGM.	FINAL WTGM.	% WT. Loss	Notes
-	25HNO3-5HF-5wT. % CITRIC ACID	R. T.	က	M I N.	3.4716	3.2437	9*9	RAPID REACTION & STAINING
വ	50HN03-2HF-5 LATIC ACID	R. T.	0.5	Σ	ı	ı	ı	RAPID REACTION & STAINING
4	25HNO3-5HF-5 SATURATED CRO3 SOLUTION	R. T. 135 120	5 0 0	Σ Σ Σ Ξ Ξ Ξ Ξ Ξ Ξ	3.1800 3.4898 .0541	3.1800 3.4860 .0257	0 .11 52.4	(B) NO APPRECIABLE OXIDE REMOVAL 1"X.5" .002" PURE TI SPECIMEN
ω	30HNO3-10HF-5 SATURATED CRO3 SOLUTION	R. T. 100 90	0000	; ; ; ; ; ; ; ; x x x x	3.4183 3.4170 3.4860 .0535	3.4124 3.4120 3.4791 .0355	.14 .16 .20 .33.7	(B) NO APPRECIABLE OXIDE REMOVAL 1"X.5 :002" PURE TI SPECIMEN
(c) S	95H2SO4-4.5 HN035HF-18.8 GM/LITER CRO3	120 120	വത	Z Z Z Z	3.4765 3.4700	3.4765 3.4697	0.01	(B) NO APPRECIABLE OXIDE REMOVAL
(o) 3	95H2SO4-4.5HF5HNO3- 2 WT. \$ CRO3	120	ო	ž E	3.4189	3.4187	900•	
7	A: 10 WT. % NAOH-5 WT. % KM _N 04 85 WT. % H20	90+10	വ	Σ Σ	3.3724 3.4360	3.3556 3.4207	4.5 5	Excellent cleaning Excellent cleaning
	B: 15 cc. H2S04-15 cc HCL- 70 cc H20-12 GM. CR03	1204	ιΩ	ż Z	3.4697 1.0403	3.4550 1.0160	2.3	(B) EXCELLENT CLEANING 1"X1"X.5" HONEYCOMB SAMPLE (1/4" CELL X .002" FOIL) DIFFUSION BONDED WITH A .002" PURE TI-INTER-
(A) ALI	(A) ALL CLEANING PROCEDURES PRECEDED BY A TRICHLORETHYLENE VAPOR DEGREASE AND ALKALINE CLEANING (6-10 07/64)	Y A TR	CHLO	RETHYL	ENE VAPOR	DEGREASE	AND ALK	MEDIATE - EXCELLENT CLEANING ALINE CLEANING (6-10 02/GAL

(A) ALL CLEANING PROCEDURES PRECEDED BY A TRICHLORETHYLENE VAPOR DEGREASE AND ALKALINE CLEANING (6-10 OZ/GAL WYANDOTTE WLG FOR 5 MIN. AT 180°F)
(B) SPECIMEN PRE-OXIDIZED 5 MIN. AT 800°F IN AIR
(C) CLEANING PROCEDURE USED IN ASD-TDR-62-937
(D) CLEANING PROCEDURE USED BY FANSTEEL METALLURGICAL CORP.

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TABLE 2

RECOMMENDED PRE-BRAZE CLEANING PROCEDURES

TZM CLEANING PROCEDURE

- 1. VAPOR DEGREASE
- 2. ALKALINE CLEAN 6-10 oz./GAL. WYANDOTTE WLG AT 180 + 10°F FOR 5 TO 10 MIN. OR EQUIVALENT ALKALINE CLEANER
- 3. COLD TAP WATER RINSE
- 4. ALKALINE ETCH

10 WT. % NAOH

5 WT. % KMNO4

85 WT. % H20

Use 5 min. at $90 \pm 10^{\circ}$ F for core and facings

- 5. COLD TAP WATER RINSE
- 6. SMUT REMOVAL

15 cc. H₂SO₄ (96%)

15 cc. HCL (38%)

70 cc. H₂0

12 gm. CHROMIC ACID

Use 5 MIN. AT 120 + 10°F ON CORE AND FACINGS

- 7. COLD TAP WATER RINSE
- 8. DISTILLED WATER RINSE
- 9. ALCOHOL DIP (MANDATORY ON CORE OPTIONAL ON FACINGS)
- 10. AIR DRY

D-36, Pure TITANIUM, AND TI-13V-11CR-3AL CLEANING PROCEDURE

- 1. VAPOR DEGREASE
- 2. ALKALINE CLEAN 6-10 oz./GAL. WYANDOTTE WLG AT 180 + 10°F FOR 5 TO 10 MINUTES OR EQUIVALENT ALKALINE CLEANER
- 3. COLD TAP WATER RINSE

TABLE 2 (CONTINUED)

ساده د سید د

4. ACID ETCH

2.0 vol. % HF (50%)

25.0 vol. % HNO3 (70%)

73.0 VOL. % H20 (DISTILLED)

Use 10 minutes at room temperature on D-36 facings.

Use 5 MINUTES AT ROOM TEMPERATURE ON D-36 CORE.

Use 1 to 3 minutes at room temperature on Ti and Ti-13V-11Cr-3AL BRAZE FOILS.

- 5. TAP WATER RINSE
- 6. DISTILLED WATER RINSE
- 7. ALCOHOL DIP (MANDATORY ON CORE, OPTIONAL ON FACINGS AND BRAZE FOILS)
- 8. AIR DRY

TABLE 3

RECRYSTALLIZATION DATA FOR .002" TZM FOIL AND .011" TZM SHEET

EET

Note: All specimens heated at approximately 650°F/min. To temperature and cooled below 1500°F within one minute in 0.2 micron or better vacuum.

TABLE 4

BRAZING ENVELOPE COATING EVALUATION DATA

.012" SPECI-	PURE TITANIUM COATING		Time To Failure Min.	*Hardness K hn	Notes
TA TB TC	Uncoated Uncoated Uncoated	2400 2600 2900	9.3 3.5 .9		TITANIUM EMBRITTLED TITANIUM EMBRITTLED TITANIUM EMBRITTLED
TD TE TF	UNCOATED UNCOATED UNCOATED	2400 2600 2900		C 91	SPECIMENS HELD 2 MIN. AT TEMP. IN ARGON AND EX-HIBITED EXTREME GRAIN GROWTH & "ORANGE PEEL" - ALL SPECIMENS DUCTILE AFTER THERMAL EXPOSURE
T I T7 T2	.004" + .001" AL ₂ O ₃ -2.5TIO ₂ PLUS .002" + .001" PURE AL	2400 2400 2600	10+ 10+ 10+	C 506 E 565 C 480	TITANIUM EMBRITTLED
T5 T3 T4 T8		2600 2900 2900 2900	10+ 1.2 1.1 3.9	E 494 C 877	TITANIUM EMBRITTLED TITANIUM EMBRITTLED TITANIUM EMBRITTLED TITANIUM EMBRITTLED
T11	.004" + .001"	2400	10+	C 534	TITANIUM EMBRITTLED
T12	AL203-2.5T102 PLUS PURE AL	2600	10+	E 675 C 467 E 492 C 976	TITANIUM EMBRITTLED TITANIUM EMBRITTLED
T13	L	2900	5.9	E 948	TITANIUM EMBRITTLED
T15	.008" + .001"	2400	10+	C 422 E 564	TITANIUM EMBRITTLED
T16	AL203-2.5TIO2 PLUS .002" + .001" PURE AL	2600	10+	C 765 E 790	TITANIUM EMBRITTLED
T17 T18		2900 2900	3.0 8.4	C 560 E 940	TITANIUM EMBRITTLED TITANIUM EMBRITTLED
TG	.004" AL ₂ 0 ₃ -2.5T10 ₂	2400		C 414 E 467	SPECIMENS HELD 2 MIN. AT TEMP. IN ARGON - TITANIUM EMBRITTLED
TH	.004" ZRO2	2400			SPECIMENS HELD 2 MIN. AT TEMP. IN ARGON - TITANIUM EMBRITTLED
TJ	Brutcher Co. Frit No. GM-875	2400			SPECIMENS HELD 2 MIN. AT TEMP IN ARGON - TITANIUM EMBRITTLED

TABLE 4 (CONTINUED)

.012	" PURE TITANIUM	T	TIME TO		
SPEC MEN	COATING		FAILURE MIN.	*Hardness KHN	Notes
TK	GM-876	2400			Specimens Held 2 Min. AT
TL	GM- 877	2400			TEMP. IN ARGON - TITANIUM EMBRITTLED
TM	Ferro Corp. Frit No. R707	2400			Specimens Held 2 min. AT TEMP. IN ARGON - TITANIUM EMBRITTLED
TN	159-1	2400			Specimens Held 2 min. AT TEMP. IN ARGON - TITANIUM EMBRITTLED
T22	.004" Cr plus .0035" Al	2900	.7		SPECIMEN FAILED DUE TO INCIPIENT MELTING
T28	.003" PURE CR PLUS	2400	10+	C 109 E 113	TITANIUM DUCTILE
T26	.003" PURE AL	2600	10+	C 217	TITANIUM SLIGHTLY EMBRITTLED
T27		2900	1.3	E 165	TITANIUM SLIGHTLY EMBRITTLED
T29		2900	3.75		TITANIUM SLIGHTLY EMBRITTLED
T30		2900	.6		TITANIUM SLIGHTLY EMBRITTLED
T31	.006" PURE CR PLUS	2600	10+		TITANIUM SLIGHTLY EMBRITTLED
T32	.003" PURE AL	2600	10+		TITANIUM SLIGHTLY EMBRITTLED
T33		2900	.6		TITANIUM SLIGHTLY EMBRITTLED
T34		2900	.6		TITANIUM SLIGHTLY EMBRITTLED
T35	.003" PURE CR PLUS	2900	.1		TITANIUM SLIGHTLY EMBRITTLED
T47	.003" MoSi2	2900	10+		TITANIUM EMBRITTLED
T48	.003" MoS12	2900	5.1+		TITANIUM EMBRITTLED
T49	.003" MoSı2	2900	1.5		TITANIUM EMBRITTLED
T39	.006" MoSI2	2600	10+		TITANIUM EMBRITTLED
T40	.006" MoSI2	2900	1.45		TITANIUM EMBRITTLED
T41	.006" MoSI2	2900	4.0		TITANIUM EMBRITTLED
T50	.006" MoSI2	2900	1.5		TITANIUM EMBRITTLED

TABLE 4 (CONTINUED)

.015"	F82				
SPECI-	COATING	Test T Temp FA		*Hardness KHN	Notes
C1	.004" AL203-2.5T102	2900			SPECIMENS HELD 2 MIN. AT TEMP IN ARGON - F82 WAS DUCTILE
C2	.004" AL203-2.5T102	3200			Specimens Held 2 min. AT TEMP. IN ARGON - F82 WAS DUCTILE
C3	.004" ZRO ₂	2900			Same as C1 and C2
C4	.004" ZRO2	3200			Same as C1 and C2
.029"	F82				
C5	.004" AL ₂ 0 ₃ -2.5TI0 ₂ PLUS .003" PURE AL	2900	4.6		F82 EMBRITTLED
C6	.004" AL ₂ 0 ₃ -2.5T10 ₂ PLUS .003" PURE AL	3200	3.6		F82 EMBRITTLED
C7	.004" AL203-2.5T102	3200	4.3		F82 EMBRITTLED
C13	.006" AL203-2.5T102	3200	.6		F82 EMBRITTLED
C14	.006" AL203-2.5T102	3200	.55		F82 EMBRITTLED
C15	.006" AL203-2.5T102 PLUS .003" PURE AL	3200	2.0		F82 EMBRITTLED
C16	.006" AL ₂ 0 ₃ -2.5T10 ₂ PLUS .003" PURE AL	3200	1.5		F82 EMBRITTLED
C8	.003" PURE CR PLUS .003" PURE AL	2900	9.1		F82 EMBRITTLED
C9	.003" PURE CR PLUS .003" PURE AL	3200	2.2		F82 EMBRITTLED
C10	.003" PURE CR PLUS .003" PURE AL	3200	1.2		F82 EMBRITTLED
C11	.003" MoS12	2900	. 1		F82 EMBRITTLED
C12	.003" MoS12	2900	2.2		F82 EMBRITTLED

^{*} C = MICROHARDNESS AT CENTER E = MICROHARDNESS AT EDGE

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